PRODUCTION OF BIODIESEL FROM FOOD WASTE

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by

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under the supervision of

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This is to certify that the work presented in the dissertation entitled "Production of biodiesel from food waste" submitted by Sipra Barik, Roll Number 614CE1001, is a record of original research carried out by her under my supervision and guidance in partial fulfillment of the requirements of the degree of M.Tech (Research) in Civil Engineering. Neither this dissertation nor any part of it has been submitted earlier for any degree or diploma to any institute or university in India or abroad.

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DEDICATION

I dedicate this thesis, to my parents and friends. Thanks for your love and encouragement during this journey. I would also like to thank my brother for his encouragement.

Sipra Barik

DECLARATION OF ORIGINALITY

I, Sipra Barik, Roll Number 614CE1001, hereby declare that this dissertation entitled "Production of biodiesel from food waste" presents my original research work carried out as a M.Tech (Research) student of NIT Rourkela and, to the best of my knowledge, contains no material previously published or written by another person, nor any material presented by me for the award of any degree or diploma of NIT Rourkela or any other institution. Any contribution made to this research by others, with whom I have worked at NIT Rourkela or elsewhere, is explicitly acknowledged in the dissertation. Works of other authors cited in this dissertation have been duly acknowledged under the section "Reference". I have also submitted my original research records to the scrutiny committee for evaluation of my dissertation.

I am fully aware that in case of any non-compliance detected in future, the Senate of NIT Rourkela may withdraw the degree awarded to me on the basis of the present dissertation.

March, 2017

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ABSTRACT

Food waste generated faces serious environmental problem. It pollutes surrounding land, air and water. There is an urgent need of its sustainable disposal technique. In this study, an innovative initiative has been taken to chemically characterize the food waste and identify the possible reuse and disposal techniques. Food waste samples are collected from kitchen of girls hostel of National Institute of Technology, Rourkela. Collected food waste samples were dried by various methods i.e., oven drying (55 ºC, 75 ºC, 105 ºC), freeze drying (-4 ºC) and sun drying. It is observed that oven drying method at 105 °C is the optimum temperature for maximum dewatering of food waste. The dried samples were further used for lipid extraction using solvents. Lipid profile analysis of the food waste samples were performed using a combination of different times (20, 40, 60, 80 and 100 min) of contact with varying solvent to food waste volumes (1:4, 1:2, 1:1, 2:1, 3:1 and 4:1). Gas chromatography- mass spectrometry (GC-MS) and gas chromatography- flame ionization detector (GC-FID) analyses have been performed to identify and quantify the presence of organic compounds in lipid from food waste. The results obtained from lipid profile analysis suggest that the kitchen food waste can be an innovative raw material for biodiesel production. The extracted lipid was transesterified for biodiesel production. During transesterification, effect of temperature, reaction time and methanol: lipid ratio was performed to study its effect on biodiesel production. Conversion of lipid to biodiesel increases with temperature upto the boiling point of methanol then it decreases. The produced biodiesel was analyzed in GC-FID for FAME analysis and compared with international standards. Chemical characterization (Ca, Mg, Cu, Zn, Cr, Fe, Ni) have also been performed of residue left after lipid extracted from food waste to identify its other possible reuse techniques. Calcium (20.36 mg/kg, iron (30.84 mg/kg), magnesium (3.00 mg/kg) and chromium (1.28 mg/kg) dominate the metallic species. The residue thus can be further reused by pharmaceutical industries after extracting these metals and also as soil and plant nutrients.

Keywords: biodiesel, characterization, food waste, lipid, transesterification

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CHAPTER 1

INTRODUCTION

1. INTRODUCTION

During the past few decades, waste generation problem has increased due to urbanization and industrialization especially, solid waste. The term solid waste means any material that is either in solid or in semi solid state. Major categories of solid waste include municipal solid waste (MSW), agricultural wastes, industrial wastes and hazardous wastes. Municipal solid waste represents a small fraction of total solid waste but gets more attention because people and environment are affected by its subsequent management (Goel, 2008). In 2002, global solid waste generation was as 12 billion tones out of which MSW generation was only 1.6 billion tones. The study by World Bank estimated that the worldwide MSW may increase from 1.3 billion tons/year (2012) to 2.2 billion tons/ year (2025) (World Bank, 2012). In India during 2015, average MSW waste generation was 700 tons per day (Pandey et al., 2016). Urbanization, industrialization and economic development in developing cities and countries have led to generation of huge amount of MSW both in quantity and composition. In most of the developing countries this has become a major concern for government departments, pollution control agencies and public (Glawe et al., 2005; Erdogan et al., 2008).

Municipal solid waste generated contains a considerable amount of food waste. Several billion gallons of food waste are produced from kitchens of residential societies, restaurants, hostels, canteens and food and meat processing industries. The national waste report of Australia published in 2008 estimated that food waste produced approximately one-third of MSW and one-fifth of commercial and industrial waste streams. Food waste generated in Australia amount to 7.5 million tones in year 2008 (Mason et al., 2011). According to the report given by Hong Kong environment bureau, 9,000 tons of MSW thrown away everyday at landfills, 40% of it consists of putrescible. Approximately, 90% putrescible is food waste (Karmee and Lin, 2014). Other developed countries like Taipei and Seoul produce 1,82,000 tons per year and 7,67,000 tons per year of food waste, respectively. In characterization of municipal solid waste, India comprises maximum of food waste (31.9%) as compared to other wastes such as plastic, textile, paper, glass, cardboard, ash, leather and metal waste (Srivastava et al., 2014).

Everyday billion gallons of food wastes generated from different sites are collected and thrown in an open area or dumped in landfill without proper disposal technique in countries like India. Landfill disposal practice has created local nuisance and pollution to human health such as bad odour, air pollution and leaching (Karmee and Lin, 2014). Food wastes affect adversely to climate, water bodies, land and biodiversity and also produce harmful gases like

carbon dioxide, greenhouse gases (Karmee and Lin, 2014). Landfill disposal also require a lot of space that is further a problem for metropolitan cities because of land dispute. To overcome mentioned problem proper management with food waste is a crucial step.

For reduction of volume and maintenance problem, recycling of food waste is necessary (Karmee, 2016). There is an urgent need to recycle and reuse of food wastes generated from different sites that are collected and thrown in an open area or dumped in landfill without proper disposal technique (Karmee and Lin, 2014). Food waste can be used as composting. In Korea, Pusan metropolitan city has introduced effective microorganisms (EM) into food waste for composting few years back. Compost produced from these microbes will improve anti-oxidant capability of soil and makes the soil more fertile. This waste reduction technique will prevent putrefaction and reduces offensive odor produced from food waste disposal (EM Research Organization, 1995; Higa, 1996).

Food waste can also be used as yellow grease for diesel fuel engines. The United States restaurants provide food waste for fuel production (Saillant, 2003). By using anaerobic digester food waste is converted to energy. This digester use series of processing stages where small organisms break down biomass into methane gas that can further be used to power electric generators (Gracia, 2011). Food wastes can also be valorized for energy production by using some common techniques such as composting, recycling and incineration (Karmee and Lin, 2014).

1.1 BACKGROUND AND MOTIVATION

Rapid urbanization and industrialization led to increase of fuel consumption resulting in depletion of fossil fuel, imposing energy crisis in near future. Till now, energy crisis problem has been met by fossil fuel which is a limited resource. Moreover, depletion of fossil fuel has led to fuel price increase and affect environment by producing greenhouse gases, ozone depletion, eutrophication, acidification, deforestation etc. As fossil fuel is a limited source of energy, there is an urgent need to search for an alternative resource that would be economically efficient, socially equitable and environmental friendly (Elsolh, 2011). Worldwide energy consumption has also increased in the last few decades. In year 2004, fossil fuel consumption is 78% out of total primary consumption. This includes 32.8% oil, 21.1% natural gas and 24.1% coal (Beretta, 2005). Most of the energy is consumed by transportation and industry sector.

During the year 2013-14 total diesel and petrol consumption was 69,080 trillion metric

tons (TMT) and petrol was 15,744 TMT. Out of total fuel consumption, diesel (44%) consumption is more as compared to petrol (10%). As fossil fuel is a limited resource, its price is expected to rise in near future and combustion of these fuels causes severe environmental problem. Scarcity of fuel and negative effect on environment has led to search for an alternative renewable resource that will be eco-friendly (Hill et al., 2006).

To overcome these problems, biodiesel production from food waste is one of the innovative ways to utilize food waste i.e., food waste which has zero value can be converted to high value added product. Recycling of food waste will increase energy production, environmental friendly and lower space requirement for landfill. Pure or blended from of biodiesel can be used in any diesel engine. No modification in engine is required, and hence less-expensive, renewable, clean-burning fuel. Biodiesel is the most promising fuel to replace an appreciable amount of petroleum based diesel fuel. It is produced from raw materials like vegetable oil, animal fats, plant oil or any lipid containing source which is renewable and biodegradable. It does not increase carbon dioxide level in the atmosphere and minimize the effect of greenhouse gas (Vicente et al., 2004; Antolin et al., 2002). In terms of sulphur content, flash point, aromatic content and biodegradability, biodiesel is better than diesel fuel (Martini and Schell, 1997). Biodiesel is fatty acid methyl ester (FAME) produced by transesterification of lipid or oil source in presence of acid, base or enzyme as catalyst (Siddhique and Rohani, 2011).

1.2 HISTORY OF BIODIESEL

Rudolf Diesel invented the diesel engine in 1893 and ran it on fuel produced from peanut oil. People were interested on petroleum product due to its easy availability and at cheaper rate (Greg, 2005). In 1930s, researchers were interested to separate glycerin and fatty acids produced from vegetable oil. Belgian patent was granted to G. Chavanne in 1937, for an ethyl ester of palm oil which is today known as biodiesel. In 1938, a passenger bus fueled with palm oil ethyl ester and travelled between Brussels and Louvain (Gerhard Knothe, 2005). During World War II (1939 to 1945), due to discontinuous supply of petroleum countries like Brazil, Argentina, China, India, and Japan used vegetable oil. After the end of world war, petroleum products were again cheap resulting in decrease in demand of vegetable oil fuel.

Diesel engine used early was designed to run on fuels produced from kerosene and coal dust and had complex injection systems. It was only a matter of time before someone could identify the potential of vegetable that would make excellent fuel because of their high

energy content. The first public demonstration of oil based diesel was at 1900 world's fair when the French company commissioned the Otto Company to build a diesel engine that would run on peanut oil. Rudolf diesel later did extensive work on vegetable oil fuels and became a leading proponent of such a concept, believing that farmers could benefit from providing their own fuel. However, it almost took a century to implement such an idea to reality. After the death of Dr. Diesel in 1913, petroleum was available worldwide in variety of forms which is today known as diesel fuel. Due to availability of petroleum at cheaper rate, the diesel engine was changed to match the properties of petroleum diesel fuel. For the next 80 years diesel engines would become the industry standard where power, economy and reliability are required.

1.3 ADVANTAGES AND DISADVANTAGES OF BIODIESEL

Biodiesel is a fuel that has gained a lot of public attention because it is environmental friendly and is highly appreciated by world. It is non-toxic, biodegradable and renewable fuel. Although biodiesel has gained much importance in recent years, it has few disadvantages also. Biodiesel when compared to petro diesel more amount of biodiesel is required. Some of advantages and disadvantages are discussed below.

1.3.1 Advantages of Biodiesel over Petroleum Based Diesel Fuel

Biodiesel has many advantages. Biodiesel, B100 (100% biodiesel without blend) can reduce CO_2 emission by 78% and carcinogenic properties emitted from diesel fuel by 94% (National Biodiesel Board, 2006). According to EPA, degradation rate of biodiesel is four times faster as compared to conventional diesel fuel. Biodiesel also reduces dependency on oil imported from other countries leading to increase in energy security. It acts as lubricant, which helps to remove dirt from the engine that may get clogged and cause engine failure. Biodiesel blend of 1% could increase the lubricity of fuel by 65% (U.S. DOE Office of Transportation Technology, 2006). Biodiesel is safer, non-toxic (toxicity is 10 times less) and has high flashpoint than diesel fuel. Transportation and storage is easier because it burns at high temperature thus reducing the chances of accident caused due to combustion.

1.3.2 Disadvantages of Biodiesel

The major disadvantages of biodiesel include high viscosity, low energy content, high cloud point and pour point, high nitrogen oxide (NOx) emissions, low engine speed and power,

injector coking, engine compatibility, and high price (Demirbas, 2008). As compared to conventional diesel fuel, biodiesel power decrease by 5% (Demirbas, 2006). The maximum torque value for biodiesel is 19.7 Nm at 1500 rpm and 21.0 Nm at 1500 rpm for petro diesel fuel. The torque value of commercial diesel fuel is greater than that of biodiesel. The specific fuel consumption values are also greater for biodiesel as compared to commercial diesel fuel. The efficiency and effective pressure values of biodiesel are also lesser as compared to diesel fuel (Canakci et al., 2006). Operating conditions such as cold flow problem, low energy content, high corrosion strip, fuel pumping problem due to high viscosity are some of the major disadvantages of biodiesel as compared to petro-diesel (Demirbas, 2007). Fuel consumption of biodiesel decreases and starts to increase with speed because power produced at low speed is low and main part of fuel is consume to overcome engine friction (Canakci et al., 2006).

1.4 TRANSESTERIFICATION

Transesterification means exchange of organic group R'' with organic group R' of alcohol. The reaction occurs in presence of acid, base or enzyme catalyst. In this reaction oil molecules break down and replace the glycerin portion of molecule with alcohol. The glycerin gets settled in the bottom layer and the upper part is biodiesel. Although transesterification is a reversible reaction but in case of biodiesel reverse reaction does not occur. This may be due to immiscible of glycerin with biodiesel resulting in separation of two phase system. The stoichiometry reaction for biodiesel is 3:1 mol ratio i.e., 3 moles of alcohol and 1 mole of oil producing 3 mol of biodiesel and 1 mol of glycerol (Encinar et al., 2005; Gerpen, 2005). The role of catalyst is to enhance the rate of reaction to convert triglyceride to biodiesel (Meher et al., 2004). Biodiesel is a clean environmental friendly, energy efficient, non-detrimental to engine performance, renewable biodegradable fuel (Van, 2005). During conversion triglyceride ester are converted to alkyl esters (biodiesel) in presence of catalyst and using methanol as solvent which produces methyl ester and methanol is replaced by glycerin. The heavier phase glycerin, settles at the bottom and lighter phase methyl ester (biodiesel) floats on top which can easily be separated by using centrifuge. This conversion of triglyceride to fatty acid methyl ester using methanol as solvent in presence of catalyst is known as transesterification. In transesterification, base catalyst when strong base is used, it is capable of deprotonating the alcohol. Strong base include sodium hydroxide or potassium hydroxides or alkoxides. The convenient method of adding catalyst to oil is dissolving the

catalyst to alcohol and then adding to oil. Dry alcohol should be added to oil because water present in alcohol will result in saponification reaction thus producing salt of fatty acids (soap) and inhibit the transesterification reaction. The carbon present on ester of triglyceride has positive charge and carbonyl oxygen has negative charge. This polarization of the C=O bond attracts the RO^- to the reaction site.

Two more RO groups present also react with other C=O groups in the same mechanism. Methanol is used because of its small size. As chain length of RO- group increases, reaction rate also increases and causes stearic hindrance. This is the reason of using methanol and ethanol in the reaction. Factors affecting transesterification reaction include moisture content, reaction temperature, free fatty acid content, oil to molar ratio, type and amount of catalyst, co-solvent and reaction time. One of the most important physical properties of biodiesel fuel is viscosity. As per ASTM D6751 specification acceptable range of kinetic viscosity at 400°C is 1.9-6.0 mm2/s. The kinetic viscosity of fatty acids found in biodiesel is affected by compound structure, chain length, position, number and nature of double bonds. High concentration of monoglycerides and diglycerides in biodiesel affects fuel quality and result in incomplete reaction during production. The free fatty acid compounds were determined by using infrared spectrometry (FT-1R), Rama spectra, NMR, gel permeation chromatography (GPC), HPLC and gas chromatography-mass spectrometry (GC-MS)(Odin et al., 2013).

1.5 NEED OF BIODIESEL FROM WASTE

Biodiesel produced from renewable resources or waste products could help to minimize fossil fuel burning problem and CO_2 production. It also helps to reduce foreign dependency for petroleum. Our transportation sector relies exclusively on petroleum for energy with its great demand for gasoline and diesel fuel. Biodiesel produced domestically from fats and vegetable oils will help to reduce our demand for petroleum. Moreover, use of fossil fuels during the past decades has led to increase of carbon dioxide (CO_2) and other green house gases that trap heat into atmosphere. Biodiesel will help to reduce green house gas emitted from transportation sector and also it is a renewable resource.

It also helps to reduce air pollution and other health related problem. According to environmental protection agency (EPA) our main motive is to reduce public health risk related to environmental pollution. Biodiesel helps to reduce emission of many air pollutants such as carbon monoxide (CO), hydrocarbons (HC), sulphur oxide (SO_x), nitrogen oxide (NO_x) and air toxics. Imports of petroleum from foreign countries take away dollars from

Indian economy. Biodiesel will help to spend the Indian economy by producing energy for our country and offer new technology for energy production to market. One billion gallon of biodiesel will provide the same benefit for B100 (100%) or in blends such as B20 (20% biodiesel with 80% diesel fuel) because the tail pipe emission of biodiesel increases with the blend level. The benefit level of any blended biodiesel can be determined by using the formula: percent biodiesel multiplied by biodiesel life cycle inventory plus percent diesel fuel multiplied by diesel fuel life cycle inventory (Sheehan et al., 1998).

1.6 OBJECTIVES OF THE STUDY

In this study, an attempt has been made to reuse food waste for production of biodiesel. The objectives of the study are highlighted below.

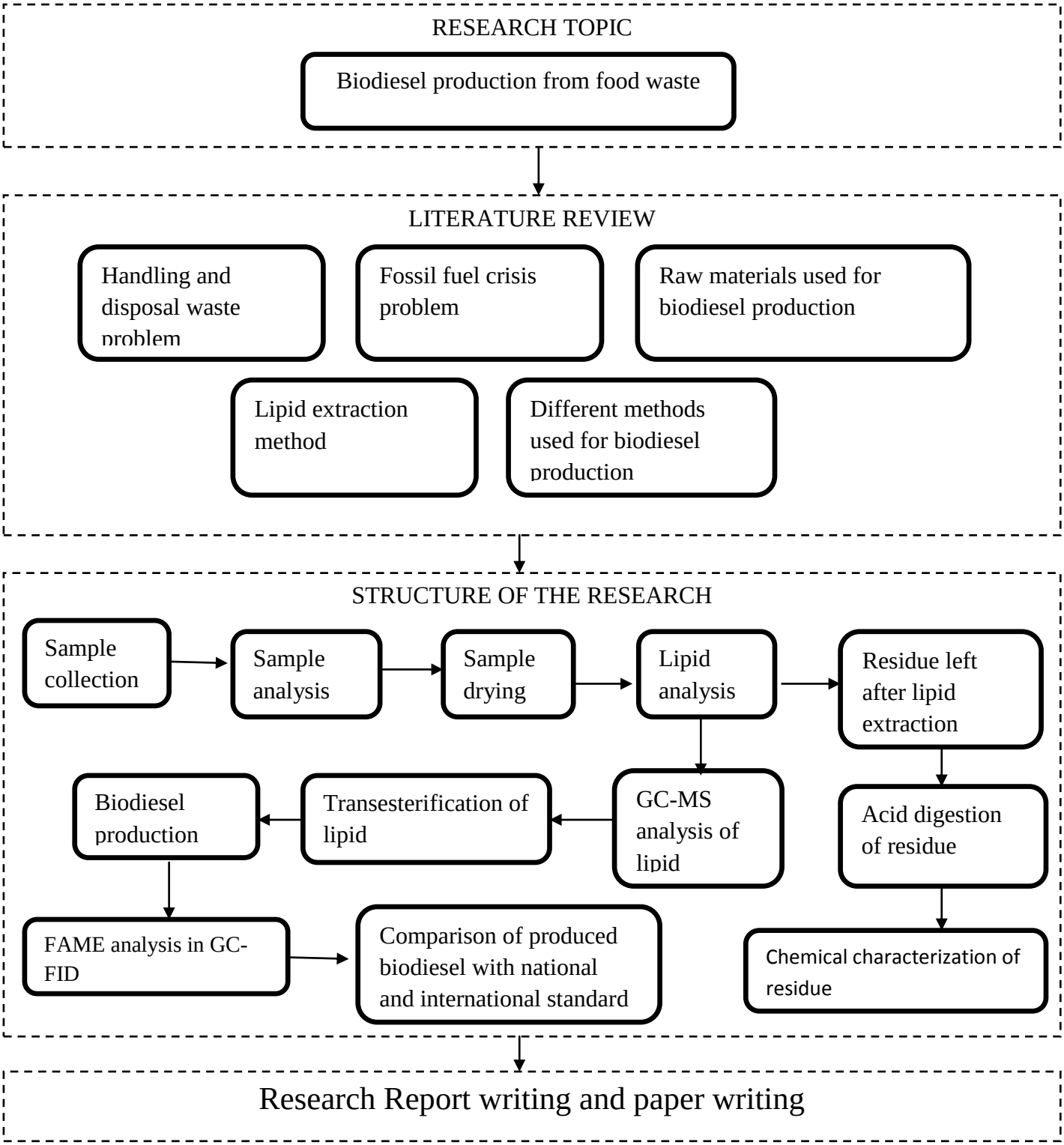
i) To extract lipids from food waste.
ii) To characterize lipid obtained from food waste.
iii) To optimize biodiesel production from food waste.
iv) To compare produced biodiesel with national and international standard.
v) To perform chemical characterization of extraction waste (residue) after lipid extraction.

1.7 STRUCTURE OF THE THESIS

Flow chart of the thesis has a subsequent sub sections. Structure of the thesis has been summarized below

- Chapter 1 describes about the reason for selection of food waste as raw material for biodiesel production. This raw material is chosen because of handling and disposal problem of food waste generation and energy crisis problem.
- Chapter 2 discuss the research and study conducted by various researchers on food waste handling and disposal problem, fossil fuel crisis problem, different raw materials used for biodiesel production and various methods opted to produce biodiesel.
- Chapter 3 describes about methods followed and materials used during the entire research work.
- Chapter 4 discuss results obtained during the research work.
- Chapter 5 describes about the conclusion and scope for further research work.

RESEARCH STRATEGY



CHAPTER 2

LITERATURE REVIEW

2. LITERATURE REVIEW

Municipal solid waste (MSW) that is generated contains appreciable amount of food waste. As food waste is a burden for the society, its handling to disposal is a major problem. Food waste contains lipids, carbohydrates, vitamins, amino acids and phosphates. Food waste containing lipids, carbohydrates and carbon can be converted to biodiesel, bioethanol and bio oil. Researchers are finding alternative and new method for biofuel production because of depletion of fossil fuel, increase demand of fuels, energy security and environmental pollution. In this regard, biodiesel has become an alternative solution. Biodiesel can be used in pure form or in blend with petro-diesel. According to the report given by food and agriculture organization of United States approximately 1.3 billion tons of food waste is disposed off at landfill worldwide without reuse. In most countries food waste is disposed of at landfill along with MSW. In present scenario various food wastes recycling technique has been developed such as anaerobic digestion, incarnation, processing for animal feed purpose, energy production and compost (Karmee, 2016). Lipid extracted from food wastes can be used to produce biodiesel (Karmee and Lin, 2014). Other than food waste some researchers (Pardo et al., 2013; Xue et al., 2006; Canakci 2007; Maceiras et al., 2010) have tried their best to produce biodiesel using different raw materials and techniques. Considering the present scenario some researchers work have been highlighted and discussed in this chapter.

2.1 DISPOSAL AND HANDLING PROBLEM OF SOLID WASTE ESPECIALLY FOOD WASTE

Lave et al. (1999) discussed about the municipal solid waste (MSW) recycling issues. In past few decades they found that closing of existing landfill and opening up new ones led to shortage of landfills. To overcome MSW disposal problem they suggested two ways i.e. energy recovery and recycling. MSW contains considerable amount of energy that could be easily recovered. Also energy recovery reduces landfill problem by 2/3rd of volume.

Ghanem et al. (2001) used kitchen food solid waste (KFSW) for anaerobic digestion by using solid phase batch system. They found that food waste contains organic fraction (fruit and vegetable) residues resulting in handling and disposal problem. To overcome this handling and disposal problem, Ghanem et al. (2001) suggested anaerobic digestion of the organic fraction as one of the possible solutions.

Nair et al. (2006) studied on organic waste produced from restaurants and canteens. These wastes were used for composting to reduce handling and disposal problem. The main

problems encountered with waste composting are its high moisture content.

Pandey et al. (2007) found that organic waste generation is high among all other component of waste. Organic carbon present in considerable proportion causes health problem. In order to avoid generation of organic waste, small community pots with revolving axis were developed along with disposal vehicles, small auto rickshaws to collect wastes.

Sharholy et al. (2008) made an attempt to study the generation, collection and transportation, characteristics, disposal and treatment technologies available for municipal solid waste (MSW) generated in India. They found that 90% of MSW is disposed of in landfill or in open dumps causing problems to human health and environment. Disposal methods include land filling, aerobic composting, vermin composting, anaerobic digestion, incineration and recovery of recyclable materials.

Khajuria et al. (2010) identified that due to urbanization and industrialization, municipal solid waste generation has increased in developing countries. In Asian developing countries, municipal corporations find difficulty in handling and disposal due to increase in amount of municipal solid waste. Waste that remains uncollected spreads on road and other public areas causing pollution.

Tweib et al. (2011) suggested food waste be used for composting. Composting has been used as a means of recycling organic matter into the soil to improve soil structure and fertility. Open dumping and sanitary landfill is a common method for food waste disposal.

Kumar and Pandit (2013) highlighted on solid waste generation in different cities of India. Solid waste contains both organic and inorganic matters. Small cities generate less waste compared to large cities. Treatment and disposal technique will help to reduce waste generation from disposal site in an environment friendly manner with little or no serious effect on health hygiene.

Njoroge et al. (2014) discussed about the current status on solid waste management (SWM). Collection rate of solid waste is about 33% out of total solid waste generated, 3.7% is recycled and rest 63% remains uncollected waste. Waste control, handling and disposal problem has been less focused which adversely affects health and environment.

Ravindran and Jaiswal (2016) highlighted that growing population led to an increasing demand for food production and large amount of food waste generation. Food waste contains complex carbohydrates, proteins, lipids and nutrients. Food waste generation causes greenhouse gas formation, foul odor, fly nuisance which can be avoided by landfill. Studies conducted by researchers showed that food waste is a renewable resource and contains lipids which can be used for biodiesel production.

2.2 FOSSIL FUEL CRISIS PROBLEM AND NEED FOR ALTERNATIVE ENERGY SOURCES

Judkins et al. (1993) found that fossil fuel accounts for about 88% of the commercial energy sources used. Fossil fuel demand has increased in recent years because they are easily available, inexpensive and people have learnt to use it efficiently. Although fossil fuel has gained popularity nowadays it will deplete soon and it causes environmental issue. The environmental problem associated with fossil fuel includes acid rain, global warming or green house gas effect. The control of emission of green house gases seems possible only by reducing the use of fossil fuel and replacing it with an alternative source that will be eco friendly and easily available.

Williamson et al. (1998) found that transport sector in UK will consume 41% of the total national primary energy demand by 2020. Reserves of petroleum and natural gas recovered economically will exhaust in next century, forcing UK to depend upon import commodities or to find an alternative to meet the energy demand. The percentage of SO_2 emitted from transport industry will also rise to 10% by 2020. The increase in energy demand by transport sector also led to emission of atmospheric pollutant like CO, NO_x and black smoke.

Dincer (2000) highlighted that the environmental problem that we face today is mainly because of pollutants emitted from transport sector. To overcome this problem, renewable energy seems to be one of the most efficient and effective solution.

Manieniyan et al. (2009) said fossil crisis can occur due market failure, over consumption, aging infrastructure and disruption in refineries that may cause restriction in fuel supply. Social and environmental problems also occur like emission of huge amount of carbon dioxide and other harmful gases, which have negative impact on environment, like sulfur dioxide. Sulfur dioxide present in atmosphere mixes with moisture and clouds in air, forming sulfuric acid also known as acid rain. When it falls causes forests, vegetation and crops to die, disturbing the equilibrium ecosystems.

Klaus (2010) said choice between energy and environment causes major energy crisis as well as environmental crisis or both. Fossil fuel consumption emits carbon dioxide which is an unavoidable product. Therefore, fossil fuels use is directly related to global environmental concerns. 81% of the world's commercial energy supply comes from fossil fuel. At present, the carbon dioxide has risen by 2ppm per year in the atmosphere.

Janda et al. (2012) said oil crisis in 1970 led to search for an alternative i.e. biofuel and replace it with fossil fuel. Greenhouse gas (GHG) reduction was one of the major concerns

for government public policies supporting the wider use of biofuels.

Gera et al. (2013) said India's energy demand continues to rise despite of global financial crisis. Energy consumption in India is more in residential, agricultural and commercial purposes compared to China, Japan, Russia, EU-27 and US. Non renewable energy obtained from natural processes will get exhausted very soon. Promoting renewable energy as a replacement of fossil fuel is a better option taking into account countries energy security and economic growth.

Chinnammai (2014) said growing energy consumption has resulted in country dependency on fossil fuels such as coal, oil and gas. Due to rise in fuel price of gas and coal and shortage of fossil fuel in future has led to think about energy security and sustain country's economic growth. Fossil fuels use has also led to increase in environmental problems both locally and globally. Due to this reason, there is an urgently need to develop a renewable fuel that can replace fossil and help to develop sustainable path.

2.3 RAW MATERIALS USED FOR BIODIESEL PRODUCTION

Ali and Hanna (1994) used vegetable oil as a raw material for biodiesel production to replace diesel fuel. Fuels derived from vegetable oil provide good engine performance in short term. However, by various processing methods vegetable oil can modified to be used as a diesel fuel. Esterification is one of the possible ways to modify the oil and reduce its viscosity. However, vegetable oil processing cost can be reduced by improving the plant capacity and it would become more viable alternative for diesel fuel.

Predel and Kaminsky (1998) conducted study on rapeseed oil to examine if the valuable contents of the grain can be used for biofuel production. At 700 °C aromatics were the most important compounds in the oil fraction which was little bit less found at 600 °C. It was concluded that rape seed can be pyrolysed to give fatty acid derivatives at low temperature (500-600 °C) and aromatic compounds at higher temperature (700 °C). In pyrolyis method approximately 40% of oil can be obtained.

Ma and Hanna (1999) found that many researchers have used vegetable oil for diesel fuel production. Vegetable oil includes palm oil, soybean oil, coconut oil and rapeseed oil. Animal fat have also been used instead of vegetable oil. Methods applicable to vegetable oil are not applicable to animal fats. This may be because of natural property differences. Microalgae have also been found as a source of methyl ester diesel fuel engine. Some natural glycerides contain high level of unsaturated fatty acids but their direct use is restricted

because of high viscosity.

Cardone et al. (2003) used brassica Carinata as an alternate oil crop for biodiesel production in Italy. Brassica carinata is a native plant of Ethiopian highlands mainly used as food by Ethiopians. The transesterification of oil has been performed for biodiesel production extracted from the B. carinata seeds and examined for physico-chemical properties suitability to be used in diesel.

Demirbas (2005) used vegetable oil for biodiesel production via both catalytic and non catalytic method. Out of the studied vegetable oils, sunflower, safflower, soybean, cottonseed, rapeseed and peanut oil are identified and considered as alternative raw material for biodiesel production to be used in diesel engine. Dilution, micro-emulsification, pyrolysis and transesterification are the techniques to reduce the high viscosity of oil. High viscosity causes poor atomization of fuel spray and less fuel injection.

Xue et al. (2006) derived a new method for preparing raw material for biodiesel production. Monosodium glutamate wastewater after dilution was well treated to form fermentation broth to biosynthesize lipid as a raw material for biodiesel production. Adding glutamate to foods increases their quality, acceptability and consumption. It is expected to produce a cheaper raw material from an organic wastewater through microorganism fermentation.

Canakci (2007) found that waste cooking oil has a great potential to extract lipid for biodiesel production. Biodiesel produced from food-grade vegetable oils is more expensive than diesel fuel. Therefore, it is not economical and feasible to produce biodiesel from food-grade vegetable oil. Transesterification process cannot be applied directly to restaurant waste oils and animal fats because of presence of high free fatty acid (FFA). Therefore, acid catalyst is added to reduce FFA level of oil.

Gouveia and Oliveira (2009) suggested that oleaginous crops such as rapeseed, soybean, sunflower and palm have potential for biodiesel production. However, biodiesel can also be produced from microalgae. Microalgae was found to be a suitable feedstock because of high oil content that could be extracted, processed and refined to produce transportation fuel by using current available technology. These microalgae grow at faster rate, use non-arable land and non-potable water, require less water and do not affect food crops cultures.

Rajvanshi et al. (2012) studied the potential of microalgae to be used for biodiesel production. Some microalgae produce 80% of oil (on dry weight basis) as compared edible/ non edible oil seeds which produces 40-50% of oil. The average oil production is between 1% and 70% but under certain condition.

Ahmia et al. (2014) highlighted about the biodiesel production from renewable resources to put emphasis on use of edible oils. Edible oil which was easily available on large scale from agricultural industry all over the world produced about 95% of biodiesel. Presently, biodiesel is produced from rapeseed in Canada, soybean in US, sunflower in Europe and palm in Southeast Asia. Non edible plant oil includes jatropha, karanja, tobacco, mahua, neem, rubber, sea mango, castor, cotton. Out of which jatropha, moringa and castor oils are mostly used for biodiesel production.

Karmee (2016) used food waste for liquid biofuel production by transesterification and bio-oil by Pyrolysis. Lipid extracted from food waste is tested to determine the acid value and moisture content. Base catalyzed transesterification using KOH and NaOH as catalysts is reported for the biodiesel preparation for low free fatty acid (FFA) containing feed stocks. Two- step reaction is carried out for lipid containing high amount of FFA. Acid catalyzed esterification is performed in first step and base catalyzed pretreatment is required to reduce acid value of lipid feedstock. In second step base catalyzed transesterification is performed to obtain the high yield biodiesel.

During the past centuries biodiesel were produced from vegetable oil and it proved to be better potential to replace petroleum based diesel fuel but it cannot be used directly on diesel engine due to high viscosity. To reduce viscosity of oil various methods like dilution, micro emulsion, pyrolysis and transesterification were used. Later it was found use of vegetable for biodiesel production lead to shortage of food i.e. food crisis. To overcome shortage of food and fuel crisis, other raw materials like microalgae, animal fats, waste cooking oil, sewage sludge etc were used for biodiesel production. Recently, it was observed that large amount of food waste is generated leading to handling and disposal problem. Food waste when dumped directly on land may produces foul odor and leachate. This leachate formation may affect soil structure and ground water. To minimize food waste generation problem researchers like Kiran et al. (2014) and Karmee (2016) made an attempt to produce biodiesel from food waste by transesterification and pyrolysis. They found that majority of fatty acids present in lipids produced from food waste were reported to be suitable for biodiesel production. This would help to reduce cost of production and proved to be economical as food waste has zero value cost.

2.4 LIPID EXTRACTION

Boocock et al. (1992) developed two simple and convenient methods for lipid extraction from raw sewage sludge. Methods used were soxhlet extraction and boiling extraction by using chloroform and toluene as solvent. From the data, it was found that boiling extraction method accumulate more lipid material. This method is better as it does not depend on degree of nitrogen and sulphur rejection.

Peterson et al. (1998) used rapeseed oil for biodiesel production. Past research had shown that transesterification of vegetable oil proved to be better alternative to replace diesel fuel. Transesterification of rapeseed oil was performed using ethanol in presence of base catalyst such as sodium or potassium hydroxide. The process produces two phases: heavier part glycerol and lighter part ethyl ester. Glycerol produced can be further used in industries.

Lang et al. (2001) used linseed and canola oils for biodiesel production by transesterification using potassium/ sodium alkoxides as catalyst. Ester chemical composition was determined by HPLC and fatty acid composition by GC. Based on GC analysis seven types of fatty acids were identified and all these esters were about 90% unsaturated. HPLC analysis showed that methyl, ethyl, 2-propyl and butyl esters were free from impurities. Only trace amount of impurities, mainly mono and diglycerides were identified and quantified, and all these esters were about 90% unsaturated.

Durand et al. (2004) mainly focused on studying hydrocarbons and poly aromatic hydrocarbons (PAH's). Gas chromatograph and GC-Ms analysis indicated the major components of organic fraction from petroleum derived diesel and motor oil. Result of this study clearly shows that the contaminants are mainly generated from anthropogenic petroleum sources.

Siddhique and Rohani (2011) developed the most promising renewable alternative fuel, biodiesel from various lipid sources. Experimental analyses were conducted to study the various effects of lipid extraction and biodiesel production from primary and secondary sludge using empirical models. Temperature has been considered as the most important factor for lipid extraction by using hexane and methanol as solvent. The yield of biodiesel was determined by gas chromatography.

Zhu et al. (2012) extracted sewage sludge lipid by referring national standards for food. The total processes were divided into three steps i.e., heat drying, extraction of lipids and transesterification. Solvents used for lipids extraction were hexane, ether, acetone and chloroform-methanol. Best solvent can be decided based on amount of lipid extracted.

Results show that bromopropane has higher solvent extraction efficiency as compared to toluene and n-hexane petroleum ether, while hexane-ethanol has higher efficiency in mixed solvent. Mixed solvent has higher efficiency compared to single solvent. Polar solvent can be easily extracted compared to non polar solvent.

Pastore et al. (2013) derived an alternative approach for biodiesel production from dewatered municipal sludge. Best performance of lipid was observed in terms of fatty acid methyl ester (18%) with lowest energy demand. Lipid was extracted in two step based on hexane extraction followed by methane analysis. Sulphuric acid plays a key role in the process of transesterification for glycerides formation and production of new free fatty acids from soaps.

Teo et al. (2014) extracted lipid for biodiesel production from Tetraselmis Sp.green marine microalgae. Exponential phase of microalgae was used for lipid extraction methanol-chloroform as solvent. Transesterification was performed using NaOH and enzyme catalyzed transesterification process using immobilized lipase. Identification of fatty acid methyl ester (FAME) was performed in GC and compared with FAME standard.

Karmee and Lin (2014) have found an alternative method of producing biodiesel from food waste instead of edible oil which has a direct impact on food shortage. Lipid is extracted from food waste by hydrolysis with an enzyme system accumulated in the solid state fungal culture present in edible and can be used as a potential source to produce biodiesel.

Olkiewicz et al. (2015) studied four different types of sludge (primary, secondary, blended and stabilised) for lipid extraction using hexane as solvent with acid catalyst. To improve the extraction efficiency, the pre-treatment method and duration of this treatment were investigated. However, the pre-treatment method did not significantly increase the amount of extracted lipid as well as biodiesel yield. Among the four various methods, primary sludge achieved the highest lipid and biodiesel yield 27% and 19%, respectively on the basis of dry sludge.

2.5 VARIOUS METHODS OF BIODIESEL PRODUCTION

Schuchardta et al. (1998) performed transesterification using vegetable oils and methanol as solvent. The base-catalyzed transesterification are less corrosive as compared to acid catalyzed reaction because of faster reaction rate. Base catalysts, such as alkaline metal alkoxides and hydroxides as well as sodium or potassium carbonates are mainly used in industries.

Marinkovic and Tomasevic (1998) used sunflower oil (vegetable oil) for biodiesel production by acid catalysis method using methanol and concentrated sulfuric acid. In situ transesterification method of sunflower seed oil using acidified methanol produces more fatty acid as compared to conventional reaction with pre-extracted seed oil. Acid catalysis is considerably slower and viscosity is less as compared to alkali catalysis.

Fukuda et al. (2001) highlighted on various transesterification and its suitability for biodiesel production. Acid catalysis transesterification is much slow i.e. 4000 times compared to alkali catalysis. Acid catalyzed transesterification is performed for oil/fats that have relatively high free fatty acid and water content.

Demirbas (2003) produced biodiesel from vegetable oil via catalytic and non-catalytic super critical alcohol transesterification. Dilution of oil with solvent like methanol and microemulsion of vegetable oil reduces its viscosity but engine performance still exists. Pyrolysis is less preferred compared to transesterification because it produces more biogasoline compared to biodiesel. Transesterification is performed to lower the viscosity of oil. Factors that affect transesterification are molar ratio of alcohol to oil, catalyst, reaction temperature and pressure, reaction time and FFA content in oils.

Sreeprasanth et al. (2006) said biodiesel contains long chain fatty acid alkyl esters produced by transesterification of vegetable oils or animal fats. Carbon chain with C6-C8 produces bio lubricants after transesterification. FFA present in vegetable oil reacts with homogenous acid or alkali catalyst and produces biodiesel along with glycerin. Biodiesel is produced using homogeneous mineral acid or alkali catalysts.

Ghaly et al. (2010) used plant oils, animal fats, microalgal oils and waste products such as animal rendering, fish processing waste and cooking oils as feed stocks for biodiesel production. Enzyme lipase has been used as biocatalyst for transesterification reaction for biodiesel production. Use of no/low cost waste materials as feedstock will help to reduce environmental pollution potential of wastes and produce an environmental friendly fuel.

Vyas et al. (2011) used Jatropha oil as raw material for biodiesel production using alkali (KOH) catalyzed transesterification. Four different alkaline catalysts i.e. NaOH, KOH, CH_3ONa, CH_3OK were selected for alkali-catalyzed transesterification of sunflower oil. Maximum bio-diesel yields were obtained when sodium or potassium methoxide (99.33 wt % and 98.46 wt%, respectively) was used because of the presence of hydroxide group, necessary for saponification. Biodiesel yield decreased from 91.67% to 86.71% when sodium or potassium hydroxides were used as catalysts because of the presence of the hydroxide group that forms soap by triglyceride saponification. It is easy and seems economical.

Thiruvengadaravi et al. (2012) produced biodiesel by two methods i.e. transesterification and pyrolysis. The most common method for biodiesel production is transesterification. Transesterification are mainly affected by the amount of the catalyst and alcohol, reaction temperature, pressure and time, as well as the content of free fatty acid (FFA) and water in oils. Conversion becomes complicated due to the presence of large amount of FFA (>2% w/w) in oil which form soap.

Anikeev and Yakovleva (2013) performed transesterification reactions using supercritical methanol of sunflower, rapeseed, cottonseed and cameline oils. Transesterification of vegetable oils was performed in presence of base, acid or enzyme catalysts for biodiesel production. In base catalyzed reactions, raw material and alcohol must be dewatered, because it may shift the reaction towards saponification and produces complicated product which will be difficult to separate.

Patil and Chavan (2014) used methanol and homogenous catalyst for transesterification of vegetable. The transesterification reaction was performed using both homogeneous (acid or basic) and heterogeneous (acid, basic or enzymatic) catalysts. Reaction is faster in case of homogenous base catalyst as compared to heterogeneous catalyst but separation of homogenous catalyst is costlier. In this study effort has been made to use solid acid or basic catalysts that could be use in heterogeneous catalyzed process to avoid catalyst removal and soap formation.

Ibrahim et al. (2015) discussed about the effect of high free fatty acid. Transesterification of Jatropha curcas seed oil was performed using homogenous and heterogeneous catalyst using methanol as solvent. The reaction was performed at 60 °C for 60 minutes and 3:1 moles of methanol to oil. Methyl esters content for homogenous products obtained were 95.5%, 98.4%, 97.4% and 85.7% but viscosities (0.5 mm^2/s) was less, whereas heterogeneous catalyst yielded low methyl esters of 77.9%, 73.7%, 71.1% and 76.2% but viscositiy (3.5 mm^2/s) was more.

Talha and Sulaiman (2016) performed transesterification of waste cooking oil using heterogeneous base catalyst for biodiesel production. Base catalyzed transesterification was selected because of its reusability, easy to separate from product, high reaction rate, low cost and require less energy as compared to acid catalyzed transesterification. However, heterogeneous base catalyst has certain limitations like diffusion, possibility of saponification to occur, sensitive to FFA content and produce more wastewater.

Worldwide waste generation has increased due to increase in population and industrialization. Municipal solid waste (MSW) production leads to handling and disposal

problem and affects environment. Waste that remains uncollected is found on roads producing foul smell and bad odor. Researchers have tried their best to identify the possible sustainable reuse techniques. Municipal waste generated contains a fraction of food waste. This food waste is directly dumped or disposed off on open land area that affects environment and human health. Food waste has been reused as compost and biofuel production. Biodiesel production from food waste is one of the innovative ways as it minimizes handling and disposal problem, green house gas emissions and fossil fuel crisis. Other than food waste, researchers have used raw materials like raw sewage sludge, microbial algae, vegetable oil, waste cooking oil, etc. Fats and oils present in raw materials were reused by extracting lipid. More the fat and oil content, more is the lipid yield and hence, more amount of biodiesel production. Biodiesel can be produced by various methods like pyrolysis, transesterification, dilution and micro emulsion. Among the four methods, transesterification is found to give better result. This innovative way of reusing waste for biofuel production will help to minimize handling and disposal problem as well as fossil crisis problem.

CHAPTER 3

MATERIALS AND METHODS

3. MATERIALS AND METHODS

Various experiments and experimental setup are used to identify the potential of food waste for biodiesel production and their methods are described in this chapter.

3.1 SAMPLING SITE

National Institute of Technology (NIT) Rourkela is located in Odisha, India with latitude 22° N and longitude 82° E. This institute has 12 hostels which include 9 boys hostels, 2 girls hostels and 1 married hostels. The CVR girl hostel of NIT, Rourkela with 1200 seated capacity is chosen as sample collection site. This study location is selected because of huge of amount of food waste generation. An attempt has been made to recycle and reuse a fraction of food waste thus minimizing handling and disposal problem as well as fossil fuel crisis problem.

3.2 FOOD WASTE ANALYSIS

3.2.1 Food Waste Drying Methods

About 3 kg of food waste sample has been collected from CVR girl hostel of NIT, Rourkela during peak hours (lunch and dinner) around 2.00 p.m. and 9.30 p.m. when food waste generation is predominant. Total 20 numbers of samples have been collected during peak hours for research work in an air tight container and brought to the laboratory for future analysis.

The collected food waste generated from kitchen outlet has been dried by various drying methods i.e., oven drying, freeze drying and sun drying. The main challenge to be faced for biodiesel production from food waste is an efficient lipid extraction from food waste as it contains water along with food particles and oil. As water can account upto 95-98%, in food waste so dewatering and drying constitute more than 50% of total biodiesel production cost. Thus dewatering of food waste sample is vital step for efficient lipid extraction and biodiesel production. Different drying methods has been used because according Olkiewicz et al. (2014) higher the drying temperature, lesser is the moisture content, more is the lipid extraction and hence biodiesel production. To find the optimized temperature which will be best feasible to extract lipid for biodiesel production similar drying methods have been followed for food waste drying. Therefore, the sample has been dried by various drying methods i.e., oven drying, freeze drying and sun drying. Oven drying has been performed by

hot air oven and freeze drying has been performed by Eppendorf centrifuge 5702 RH as shown in figures 3.1 and 3.2. Details of various drying methods of sample have been summarized in table 3.1.

Table 3.1: Various drying methods of food waste sample

S.N.	**Food waste drying methods**	**Temperature (°C)**	**Duration of drying (days)**	**References**
1.	Oven drying	105	1	Olkiewicz et al., 2014; Willson et al., 2010
		70	2	
		55	3	
2.	Freeze drying	-4	2	Revellame et al., 2010
3.	Sun drying	-	10	Olkiewicz et al., 2014

Figure 3.1**:** Hot air oven (oven drying method)

Figure 3.2: Centrifuge (freeze drying method)

3.3 LIPID EXTRACTION AND ANALYSIS

The extraction after drying was carried out in soxhlet apparatus using methanol as solvent as shown in figure 3.3 and Bleigh Dyer's method using methanol and chloroform (2:1) as solvent. Magnetic stirrer has been used for lipid extraction to study the percentage of lipid yield after each cycle (stage) at different times of contact and at different ratio. After extraction, solvent was removed using rotary evaporator at 70 ºC under vacuum. Then the extracted lipid was stored in desiccator overnight and weighed the next day to determine the exact extraction yield.

Figure 3.3: Lipid extraction by using soxhlet

3.3.1 Liquid- Liquid Extraction of Dried Food Waste Sample

Sequential liquid- liquid extraction was carried out in a magnetic stirrer with an agitation speed of 450 rpm, ambient temperature, using methanol and chloroform as solvent. Ten consecutive extraction stages were conducted, in which the food waste after settling was extracted again by adding fresh solvent. After each extraction stage, sample is filtered to separate the liquid and solid particles using whatman42 filter paper, 125mm (dia). Later solvent is recovered using rotary evaporator at 70 °C under vacuum pressure and reused for consecutive stage. Extracted lipids were stored overnight in a dessicator and weighed next day to determine the extraction yield. The extracted lipid was examined using gas chromatography mass spectrophotometer (GC-MS) to identify the organic compounds present in lipid and gas chromatography- flame ionization detector (GC-FID) to determine the concentration of each organic compound after identification.

3.3.2 GC-MS Analysis of Lipid

Lipid extraction was performed at different solvent to food waste ratios i.e., 1:2, 1:1, 2:1, 1:4, 3:1 and 4:1 to determine the optimized ratio. After optimization, extracted lipids have further been analysed in GC-MS (Agilent 7820A series) to identify the presence of free fatty acids. This analysis will be helpful in further step as these free fatty acids are essential component of biodiesel. Table 3.2 illustrates the respective program for GC-MS used for analysis of lipid profile.

Table 3.2: Instrumentation and Analytical conditions for the GC-MS system

Gas chromatograph	Agilent 7820A Series
Column	Agilent 122-5532E: 1 DB. 5ms 0℃-325°C (350℃): 30m x 250 μm x 0.25 μm
Oven temperature program	200℃ (5.6667min),10°C, 310°C (11.167 min)
Injector volume	1 Ml
Column flow (N_2)	1 mL/min
Pressure	17.154psi
Holdup time	1.3665 min
Inlet settlings	280℃, split ratio: 20:1
Split flow	20 mL/min

3.4 BIODIESEL PRODUCTION

Biodiesel can be produced by various transesterification processes. Transesterification process depends on the value of free fatty acid (FFA). If FFA < 1 then base catalyzed transesterification is preferred. The FFA content in feedstock should be low, because FFAs react with base catalysts to produce soap (Anikeev and Yakovleva, 2013). Base catalyst gives higher reaction rate compared to acid catalyst but base catalysts produce soap. Soap formation results in deactivation of catalyst and high production cost. Acid catalyst do not form soap and perform transesterification of free fatty acid resulting in biodiesel production (Pathak, 2015). The disadvantages of acid catalyst include slow reaction and require higher reaction temperatures. Acid catalyzed transesterification produce biodiesel from low cost raw and lowers the production cost. Base catalyzed reaction is 4000 times faster than the acid catalyzed reaction (Dholakiya, 2012). The fatty acid methyl ester obtained after transesterification were analyzed and compared with various standards to ascertain their suitability as diesel fuels.

3.4.1 Catalyst Type, Concentration and Reagent

Large numbers of efficient catalysts have been identified by researchers to enhance the reaction rate. In absence of catalyst rearrangement can be done but under certain conditions like temperature and time, leading to undesirable effects such as isomerisation, polymerization and decomposition. The most common method of producing biodiesel is transesterification by using acid, base or enzyme as catalyst. Transesterification is said to be the most favourable reaction in producing biodiesel because it can reduce the oil viscosity (Abbaszaadeh et al., 2012). Sodium alkoxides are the most efficient catalyst although sodium hydroxide or potassium hydroxide can also be used. Transesterification can be performed by using methanol in presence of sodium or potassium hydroxide as catalyst to yield fatty acid methyl ester (FAME). Base catalyzed transesterification is mostly preferred because this method produces free fatty acid methyl ester in less time (Srirangsan et al., 2009) and corrosion is less (Jayed et al., 2009). Base catalyst have faster reaction rate compared to other catalyst (Siddiquee and Rohani, 2011; Berrios and Skelton, 2008). However, reaction between FFA and alkali catalyst results in soap formation and difficulty in glycerol separation, thus lowering in biodiesel yield (Atadashi et al., 2012). More amount of catalyst is also required. Selection of catalyst is usually done on the basis of raw material and reaction condition (Kaercher et al., 2013). The important parameter affecting transesterification are methanol: oil ratio, temperature, amount of catalyst, reaction time, etc. (Odin et al., 2013). The reaction is performed at 60-70 °C close to boiling point of methanol at atmospheric pressure for given time i.e. 2.5h.

3.4.2 Transesterification of Food Waste

Transesterification of food waste was performed using acid catalyzed transesterification. Though base catalyzed transesterification is faster than acid catalyzed transesterification, it was less preferred because of soap formation and difficulty in glycerin separation. Also FAME analysis in GC did not show sharp peaks of free fatty acid methyl ester. In this study, a known amount of lipid was prepared in a round neck bottom flask as shown in figure 3.4. Solution of known amount of methanol and catalyst (H_2SO_4) was prepared. The solution was added to lipid and kept closed in an air tight flask. The flask was placed on a heating base maintaining a temperature of 65 °C close to the boiling point of methanol. The mixture was mixed continuously for 2 - 2.5 h using magnetic stirrer. Excess alcohol is normally used to ensure complete conversion of fat or oil to fatty acid methyl ester. After the formation of

methyl ester, the heating was stopped and product was cooled for some time and then it was transferred to separatory funnel. The product was left undisturbed for 24 h for complete separation of methyl ester and glycerol. The ester layer mainly contains methyl ester and methanol. Methanol present in methyl ester was recovered by using rotary evaporator at 70 °C.

Figure 3.4: Transesterification of lipid

3.4.3 Washing of Biodiesel

After transesterification, washing of biodiesel is an important step to remove catalyst, methanol and other impurities. Methyl esters present in biodiesel usually contain contaminants that affect the quality of biodiesel and need to be removed. Removal of glycerol and glycerides from biodiesel is a key step because quality of biodiesel is dependent on the content of glycerol. High concentration of glycerol in biodiesel can cause problem during storage and increase in aldehyde emissions (Faccini et al., 2011). Washing of biodiesel include wet washing and dry washing. Wet washing means adding distilled water to biodiesel. Dry washing means adding adsorbent like magnesol, anhydrous sodium sulphate, resin etc. In this study, wet washing of biodiesel has been performed after transesterification. This wet washing method is also called water wash method. Wet wash method has been selected because this is the oldest and most common method of washing. Moreover, the waste water recovered after washing can be reused for irrigation purpose after minimal treatment. Other waste treatment methods generate hazardous wastes that are difficult to disposed off. The purpose of water wash method is to remove unreacted alcohol, catalyst or glycerin present in biodiesel. It will also remove if any soap is present.

After glycerin settles in the bottom of the separatory funnel, separate the biodiesel and measure the pH of biodiesel. The pH of the unwashed biodiesel is found close to 9. Boil the

distilled water before adding to biodiesel. Slowly add warm distilled water in equal proportion to biodiesel. Allow the water to settle out of biodiesel by transferring it to the separatory funnel. It will take around 24hrs to settle down. Once the mixture has settled down, biodiesel will float on top of water. Drain off the water. After washing measure the pH of biodiesel, it should be 7. Washing can be performed multiple times to purify the biodiesel. The upper layer after separation is biodiesel and lower layer is impurities along with glycerin as shown in figure 3.5

Figure 3.5: Separation of biodiesel and impurities along with glycerin after wet washing

3.4.4 FAME Analysis

After washing, FAME was examined by gas chromatography- flame ionization detector (GC-FID).About 37 components FAME mix standard was used to identify individual FAME in the product. The program for FAME analysis has been discussed in table 3.3. After identification, FAME properties were compared with ASTM, European and Indian standard for biodiesel production.

Table 3.3: GC-FID program for FAME analysis

Column	Agilent 122-5532E: 1 DB. 5ms 0℃-325°C (350℃): 30m x 250 µm x 0.25 µm
Oven	140℃ (5 min) to 240 C at 4℃/min
Carrier gas	Nitrogen
Determination	FID
Injector	1µl, 260℃, split 100:1
Sample	Dilute to 10mg/ml in methylene chloride

3.5 CHEMICAL CHARACTERIZATION OF EXTRACTED WASTE LEFT AFTER LIPID EXTRACTION

The extracted waste left after lipid extraction has been used for acid digestion. For detailed Chemical characterization, it is necessary to digest the food wastes sample. Nearly 0.25 g of powdered samples have been taken in a digestion tube containing 4 ml of HNO_3 (65%), 2 ml of HF (40%) and 2 ml of H_2O_2 (30%). Samples have been digested by using Milestone microwave digester (MODEL START) which shown in figure 3.6.

Figure 3.6: Acid digestion of extracted waste after lipid extraction

The acid digested samples were used for chemical characterization to identify if it can be further used thus minimizing its handling and disposal problem. The samples were characterized for chemical determination following IS 3025 and using atomic absorption spectrophotometer (AA200, Perkin Elmer).

CHAPTER 4

RESULTS AND DISCUSSION

4. MOISTURE CONTENT AND LIPID YIELD

Drying of food waste sample and removal of moisture content is a vital step for lipid extraction as well as biodiesel production. The water content present may surround the solid particles present in food waste sample and inhibit the solvent to penetrate inside the solid particles. The final moisture content of the sample depends on the drying method and its temperature. It was observed that at high temperature, moisture content is low. From the table 4.1, it is observed that greater the moisture content lower is the lipid extraction.

Table 4.1: Different drying methods and its effect on lipid extraction

S.N.	Drying methods	Temperature (ºC)	Drying (h)	Moisture content %	% Lipid yield
1.	Oven drying (Olkiewicz et al., 2014; Willson et al., 2010)	105	24	0.1	37.3
		75	48	1.5	28.8
		55	72	2.4	25.4
2.	Freeze drying (Revellame et al., 2010)	-4	48	7.5	13.7
3.	Sun drying (Olkiewicz et al., 2014)	25-30	240	4.6	16.5

In general, the greater the amount of moisture contained in the sample, lesser is the amount of extracted lipids (Olkiewicz et al., 2014). Sun drying and freeze drying methods does not give best result and they are time consuming methods. Sun drying method takes about 240h for drying and it also depends on climatic condition. During rainy season sun drying of food waste sample is impossible. Also it will produce foul odor and affect environment and human health. In oven drying methods that are performed at different drying temperature of 105 °C, 70 °C and 55 °C, moisture content at 105 °C gives best result with less moisture content. Freeze drying at -4 °C takes around 48h for drying giving a moisture content of 7.5%. It was observed that among all the drying methods applied for food waste sample, oven drying method at 105 °C giving less moisture content of 0.1%. Hence, oven drying method at 105 °C is selected as the optimum temperature for drying method. Moreover, for biodiesel production long fatty acid chains are required. If heated beyond 105

°C then the carbon chain may break thus affecting biodiesel production.

The various drying methods, moisture content and its effect on lipid yield have been discussed in table 4.1. The value of lipid and biodiesel yield depends on dry weight of food waste sample. The temperature used for freeze drying (-4 °C) and sun drying method gave lipid yield of 13.7% and 16.5%, respectively showing a considerable amount of loss of lipid yield in comparison to oven drying method. The drying of sample in oven (55 °C and 70 °C) and freeze drying (-4 °C) method have also been performed. The sample was dried at low temperature to study its effect on lipid yield because according to the report given by Cordero-Esquivel et al. (1993), biomass drying by both freeze-drying and oven drying at low temperature caused an approximately 70% loss of total lipid content. It is observed that the temperature used for oven drying at 70 °C and 55 °C and freeze drying at -4°C decreased the lipid yield in comparison to oven drying at 105 °C. So, it is considered to dry sample at 105 °C temperature to prevent loss of lipid yield and efficient amount of biodiesel production. The effect of variation in lipid yield due to food waste composition has been discussed in table 4.2. It was observed that the combination of vegetarian and non vegetarian yield more lipids compared to vegetarian only.

Table 4.2: Variation of food waste composition and lipid yield

S.N.	**Days**	**Food Waste Composition**	**Lipid yield % (wt of lipid/dry wt of food waste)**
1.	Monday	Vegetarian	32.5
2.	Tuesday	Vegetarian and non-vegetarian	37.3
3.	Wednesday	Vegetarian and non-vegetarian	36.8
4.	Thursday	Vegetarian	30.2
5.	Friday	Vegetarian and non-vegetarian	36.4
6.	Saturday	Vegetarian and non-vegetarian	37.3
7.	Sunday	Vegetarian and non-vegetarian	37.2

4.1 OPTIMIZATION OF LIPID EXTRACTION FROM FOOD WASTE

The extraction of food waste was carried out using a combination of different times of contact in each stage (20, 40, 60, 80 and 100 min) and different food waste to solvent

(methanol) (2:1, 1:1, 1:2, 1:4, 3:1 and 4:1) ratio. The other operative conditions were maintained constant i.e., 30 g of food waste and 440 rpm speed at ambient temperature. The extraction of food waste was carried out at different extraction time in each stage (20, 40, 60, 80 and 100 min) and different food waste to solvent (methanol) (1:1, 1:2, 1:4, 2:1, 3:1 and 4:1) ratio. Stage means the number of cycles extraction has been carried out by for particular extraction time. In figure 4.1 it was observed that extraction of lipid yield increased in all cases with consecutive extraction stages. After sixth stage, lipid yield did not increase significantly. It was observed that lipid yield reached a constant value after eighth stage upto tenth stage indicating that no more lipids can be extracted as it has already reached equilibrium. The best value of lipid yield was achieved in last extraction stage for each ratio i.e., 1:1 (25.8%), 2:1 (37.3%), 1:2 (16.9%), 1:4 (14.9%), 3:1 (38.2%) and 4:1 (39.9%). In comparison to solvent: food waste, extraction performed at different ratios (1:1, 2:1, 1:2,1:4, 3:1 and 4:1), it was observed that best yield was obtained in ratio 2:1 with lipid yield of 37.3%. Thus, it can be concluded that lower the amount of solvent, lesser the extraction efficiency is achieved.

The contact time also plays an important role in lipid extraction. For solvent: food waste ratio of 1:4, 2:1, 1:1, 1:2, 3:1 and 4:1, the lipid yield increased as the time of extraction increased after each stage upto sixth stage. Extraction was performed for 20 min, 40 min, 60 min, 80 min and 100 min. Extraction performed for 80 min and 100 min to study if the plotted graph for different ratio is increasing after 60 min. It was observed that for 80 min and 100 min the lipid yield value was almost same as that obtained for 60 min. It can be concluded that 60 min of extraction time is enough to give best lipid yield. For 40 min of extraction time, lipid yield obtained in the ratio 1:1, 1:2, 2:1, 1:4, 3:1 and 4:1 gave extraction value of 21.3%, 16.3%, 33.3%, 12.9%, 33.7% and 35.6% respectively. The lowest lipid yield was achieved at 20 min in the ratio 1:1, 2:1, 1:2, 1:4, 3:1 and 4:1 with extraction of 19.9%, 30.6%, 15.5%, 10.9%, 32.2% and 33.8% respectively. The result obtained for 60 min in ratio 1:1, 1:2, 2:1, 1:4, 3:1 and 4:1 are 25.8%, 16.9%, 37.3%, 14.9%, 37.8% and 38.5%. This study was conducted to obtain the best extraction time for maximum lipid yield.

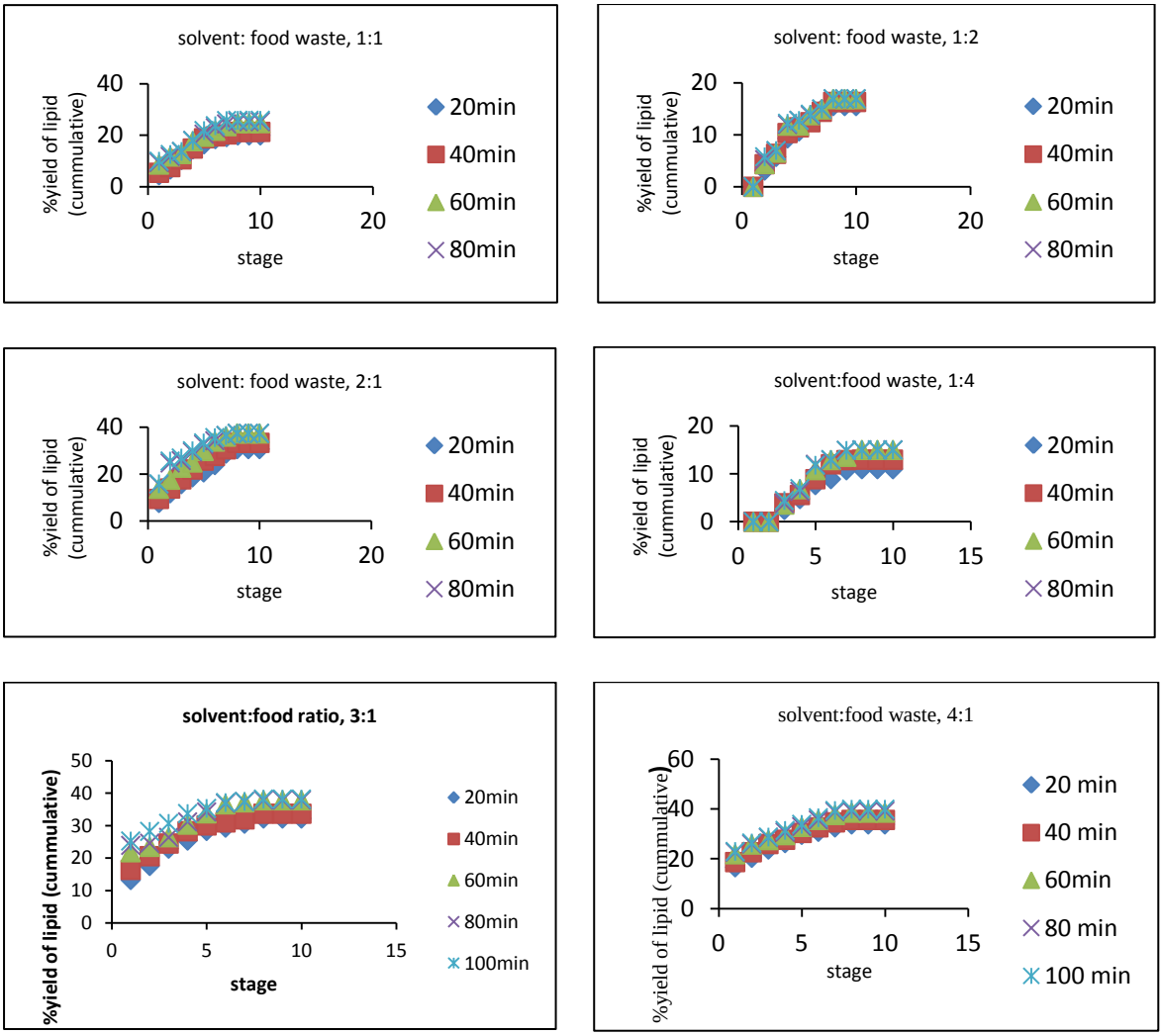


Figure 4.1: Lipid extraction in different ratio of solvent: food waste

Independent of extraction time, addition of solvent is also important factor in lipid extraction. It was observed that the lipid yield increased with the addition of solvent but upto certain stage. Beyond sixth stage there was no significant increase in lipid yield in 1:1, 1:2, 2:1, 1:4, 3:1 and 4:1. This suggests that the amount of solvent added is sufficient for lipid extraction. Also it was observed that in ratio 1:2 and 1:1, initially there was no lipid yield. It may be due to the fact that solvent added to food waste may be insufficient or the amount of solvent added may be absorbed by the food waste. Also it was observed that with minimum amount of solvent i.e. in the ratio 1:2 and 1:4 lipid yield obtained was 16.9% and 14.9% in 60 min. It was observed that for more amount of lipid extraction, more amount of solvent is required. On the other hand, for minimizing the extraction time, the best yield was obtained in 20 min in the ratio 1:1 with 19.9% as compared to ratio 1:2 and 1:4. It was observed ratio 2:1 give best lipid yield of 37.3% in 60 min at eighth stage when compared to ratio 1:1, 1:4

and 1:2 in terms of extraction time, stage and addition of solvent. When ratio 2:1 is compared with ratio 3:1 and 4:1 lipid yield in 60 min was almost same. Moreover, solvent consumption was more in case of 3:1 and 4:1 as compared to ratio 1:1, 1:2, 1:4 and 2:1. Ratios 2:1, 3:1 and 4:1 gave lipid yield of 37.3%, 37.8% and 38.5%, respectively in 60 min at eighth stage as compared to ratio 1:1 (24.7%), 1:4 (14.9%) and 1:2 (16.8%) in terms of extraction time, stage and addition of solvent. Ratio 2:1 (37.3%) gave almost same yield as that of 3:1 (37.8%) and 4:1 (38.5%). For optimized production with minimum solvent consumption of lipid yield, 2:1 ratio gave best yield of 37.3% in 60 min at eighth stage when compared to ratio 1:1, 1:2, 1:4, 3:1 and 4:1. Also ratio 2:1 gave almost same yield less solvent consumption as compared to ratio 3:1 and 4:1. Hence, ratio 2:1 is considered economical giving maximum lipid yield of 37.3% in 60 min at eighth stage.

4.1.1GC-MS Analysis of Lipid

The extracted lipids have further been analyzed in GC-MS (Agilent 7820A series) to identify the presence of organic compounds. The result shown in table 4.3 indicates the identification of fatty acids present in lipid.

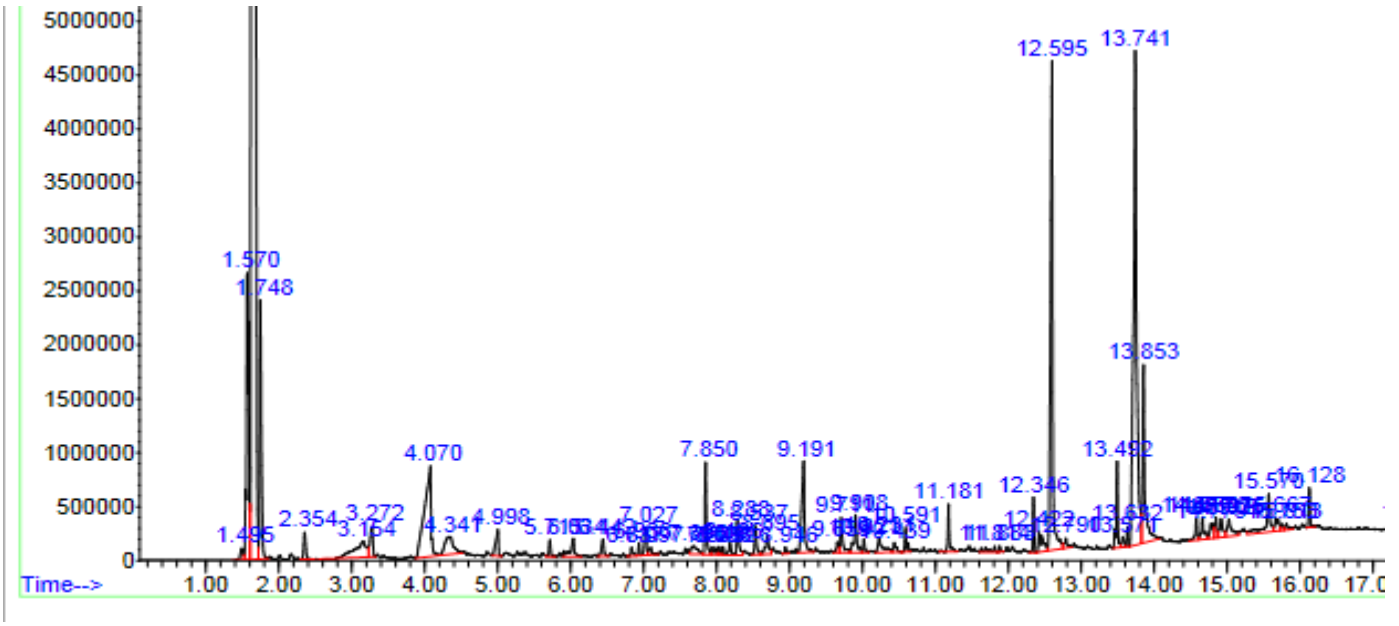


Figure 4.2: Gas chromatograph- mass spectrometry analysis of lipid

Biodiesel properties are mainly dependent on fatty acid composition. Presence of medium and long chain free fatty acids indicated its potential for biodiesel production. Free fatty acid composition of lipid profile has been shown in figure 4.2 and table 4.3. Fatty acids include

free and complex part of lipids. These fatty acids present in lipid have potential for fuel production. The chemical and physical properties of any fatty acids, such as solubility in non-polar solvents and melting point, depend on the number of carbon atoms present in molecule (Fahy et al., 2005). The higher the number of carbon atoms of the chain the higher will be melting point of the fatty acid. Fatty acids are named according to the chain length of carbon. Short-chain fatty acids, have four (C4) to ten (C10) carbons, medium-chain fatty acids have twelve (C12) to fourteen (C14) carbons, long-chain fatty acids have sixteen (C16) to eighteen carbons (C18) and very long-chain fatty acids twenty (C20) or more carbon atoms. Molecules having carbon atoms less than four i.e. C2 (acetic acid), C3 (propionic acid) are not termed as fatty acids because of high water solubility. Due to increase of double bonds in fatty acids melting point also reduces. Saturated structure having same number of carbon atoms may give rise to solid or semisolid product at room temperature while unsaturated structure may be liquid or less solid at room temperature.

Table 4.3: Free fatty acids identified in GC-MS of lipid sample

S.N.	**Retention time**	**Organic compounds**
1.	4.070	Caproic acid (C6:0)
2.	9.191	Lauric acid (C12:0)
3.	11.181	Mystric acid (C14:0)
4.	12.595	Palmitic acid (C16:0)
5.	13.741	Stearic acid (C17:0)
6.	13.853	Oleic acid (C18:0)

Lipids containing fatty acids may be both saturated and unsaturated fatty acids. Saturated fatty acids are filled containing hydrogen and have straight hydrocarbon chains with even number of carbon atoms. Fatty acids mostly have carbon atoms between C12- C22. Saturated fatty acids identified in lipid include caproic acid (6:0), lauric acid (12:0), mystric acid (14:0), palmitic acid (16:0) and stearic acid (17:0).

Unsaturated fatty acids have one carbon-carbon double bond which can be present in different positions. The most common unsaturated fatty acids contain chain length of 16-22 and a double bond with cis configuration. Cis configuration means hydrogen atoms adjacent to the double bond stick on the same side of the carbon chain. Unsaturated fatty acid identified in lipid is oleic acid (18:0). Lipid analysis is important because saturation level determines the characteristics of fat or oil. Fats and oil become more saturated by injecting hydrogen gas which bonds to available carbon. This process is called hydrogenation and

helps to make oil and fats more durable and resistant to decomposition. Hydrogenation of oil doesn't have significant effect on transesterification.

Table 4.4: Extraction of lipid by various researchers

S.N.	Researcher	Raw material	Methods adopted	Solvent	Lipid profile
1.	Boocock et al. (1992)	Municipal sewage sludge	Boiling extraction and soxhlet extraction	Chloroform and toluene	79% unsaponifiable 21% glycerides fatty acids, 45% palmitic acid, 41% stearic acid
2.	Konar et al. (1992)	Raw Atlanta dried sewage sludge (pyrolysed)	Boiling extraction	Toluene	65 wt% free fatty acid, 28 wt % unsafonifiable
3.	Durand et al. (2004)	Municipal sewage sludge	Soxhlet extraction	Chloroform -methanol	35.4% for Wissous and 11.4% for Ronchin
4.	Siddhique and Rohani (2011)	Waste water sludge (primary, secondary, blended and stabilized)	Soxhlet extraction	Hexane and methanol	-
5.	Zhu et al. (2012)	Municipal sewage sludge	Acid hydrolysis method for determination of fat in foods	Hexane, ether, acetone, chloroform -methanol	-
6.	Cheirslip and Louhasakul (2013)	Industrial waste	Soxhlet extraction	Chloroform -methanol	48.3% triglyceride, 7.8% diglycerides, 25.7% monoglycerides, 18.2% free fatty acid
7.	Pastore et al. (2013)	Dewatered sludge	Methane extraction	Hexane	-
8.	Teo et al. (2014)	Marine microalgae (tetraselmis)	-	Chloroform -methanol	Palmitic acid, stearic acid, oleic acid, linoleic acid,linolelaidic acid, linolenic acid, arochidic acid, behenic acid
9.	Olkiewicz et al. (2015)	Municipal sewage sludge	Soxhlet extraction	Hexane	Palmitic acid (31.1 to 49.4%), oleic acid (18.3 to 32.6%), stearic acid (8.3 to 15.8%)
10.	Vajpeyi and Chandran (2015)	Oleaginous microorganism	-	Methanol	Palmitic acid, oleic acid, linoleic acid
11.	Present study	Food waste	Soxhlet extraction and Bligh dyer method	Methanol and chloroform	Caproic acid, lauric acid, mystric acid, palmitic acid, stearic acid, oleic acid

Different researchers have used different raw materials and different lipid extraction methods. Some of the researchers work and the percent of lipid yield have been compared with present study lipid analysis which is discussed in table 4.4. The study identified food waste as a zero cost raw material can be used for biodiesel production thus minimizing handling and disposal problem along with fossil fuel crisis problem. It is an innovative approach to reuse zero cost material for production of high value added product. This study also controls pollution by reducing leachate generation environmental problem like foul odor and fly nuisance.

4.2 TRANSESTERIFICATION AND BIODIESEL PRODUCTION

Some researchers like Nguyen et al. (2015) and Odin et al. (2013) have used activated sludge and quassi oil as raw material for biodiesel production. They conducted experiments to study the effect of temperature, catalyst, reaction time and methanol to oil molar ratio as these factors may affect biodiesel yield. Similar experiments have been conducted with food waste to study the effect of methanol to lipid molar ratio, reaction time and temperature on biodiesel yield.

4.2.1 Effect of Methanol to Lipid Molar Ratio

In transesterification process, addition of excess amount of catalyst in lipid may produce soap giving lower amount of biodiesel yield. A fixed amount of catalyst (2.5 wt %) was added while methanol to lipid ratio was varied from 5:1 to 13:1. Reaction time was maintained for 2 hours and 30 minutes at 60-70 °C. The effect of methanol to lipid molar ratio on conversion of biodiesel is shown in figure 4.3. The maximum conversion of biodiesel was achieved in methanol to lipid molar ratio of 11:1. It is observed that the percentage of conversion of biodiesel increased from ratio 5:1 to 11:1 and then reaches constant value upto ratio 13:1. Thus to achieve maximum conversion, excess methanol is required but upto certain ratio i.e., 11:1 indicating that the amount of methanol added to lipid is sufficient for conversion of biodiesel. As transesterification is a reversible reaction, excess methanol is required to shift the reaction towards biodiesel production. Also, to break the glycerin-fatty acid linkages during transesterification for conversion of triglycerides to biodiesel, excess methanol is required. It is observed in fig 4.3 that beyond ratio 11:1, biodiesel yield attains constant value upto ratio 13:1. This may be due to dilution effect as more amount of methanol is consumed. Another reason to avoid more amount of methanol is that high methanol interferes with

separation of glycerol and biodiesel because of increase of glycerol in biodiesel as well as difficulty in washing of biodiesel and contamination of biodiesel product (Encinar et al., 2012).

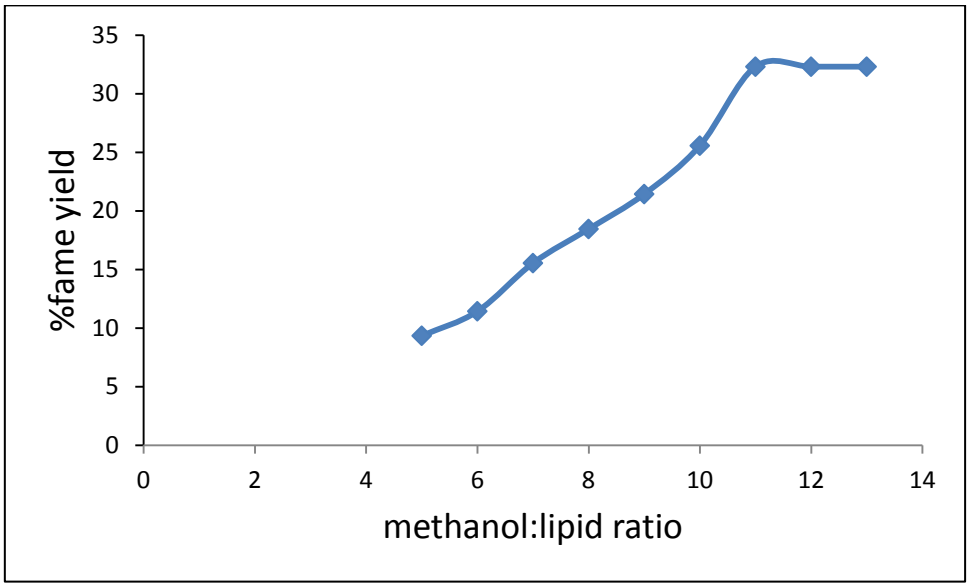


Figure 4.3: Effect of methanol: lipid molar ratio in conversion of biodiesel

Huynh et al. (2012) reported that higher methanol to lipid ratio tends to low biodiesel yield as excess methanol favors extraction of more polar compounds as well as the formation of mono-glyceride. Excess methanol produces more glycerol which increases the driving force of reverse reaction. Moreover, excess methanol increases the cost of methanol recovery. Also, minimum use of methanol subjected to reduce the cost of biodiesel production.

4.2.2 Effect of Reaction Temperature

The effect of reaction temperature on the conversion of lipid to biodiesel was studied. A fixed amount of catalyst (2.5 wt %) was taken while methanol to lipid ratio 11:1 and reaction time was maintained for 2 hours, 30 minutes. The reaction was carried at four different temperatures i.e., 50 °C, 60 °C, 65 °C and 70 °C as shown in figure 4.4. The reaction temperature of 50 °C was chosen as it is closer to the boiling point of methanol and expected to give better biodiesel yield.

Temperature is the important factor that will affect biodiesel yield. Higher reaction temperature increases reaction rate and shorten reaction time due to reduction in viscosity of lipid. From figure 4.4, it can be seen that the conversion of lipid to biodiesel increased as the reaction temperature increases. It may be due to the increase of miscibility between methanol

and lipid resulting increase of reaction rate (Amit, 2012). Miscibility of methanol and lipid are poor at room temperature. Reaction rate increases with increasing temperature resulting in increase in solubility of methanol in oil-rich phase. Higher reaction temperature results in shorter reaction time in mass transfer controlled reaction because transesterification reaction is more preferred at higher temperature (Noureddini and Zhu, 1997). Usually the transesterification should be performed below the boiling point in order to prevent evaporation of methanol. It is recommended to perform reaction below boiling point by various literatures. The maximum biodiesel yield of 32.3% was observed at 60 °C. In addition, higher temperature results in decreasing polarity of methanol and this leads to enhance the solubility of fatty acids in methanol. At or above the boiling point of methanol (65 °C), biodiesel yield decreased. This may due to the fact that methanol boils at temperature which led to evaporation of methanol and loss of the solvent, thus lowering the conversion of lipid to biodiesel. Kusdiana and Saka (2004) also reported that in supercritical state, methanol acts as an acid catalyst in transesterification reaction. Temperature increases the energy of the reacting molecule and also improves the miscibility of alcoholic polar media into a non-polar oily phase resulting in much faster reaction (Ogbu and Ajiwe, 2013).

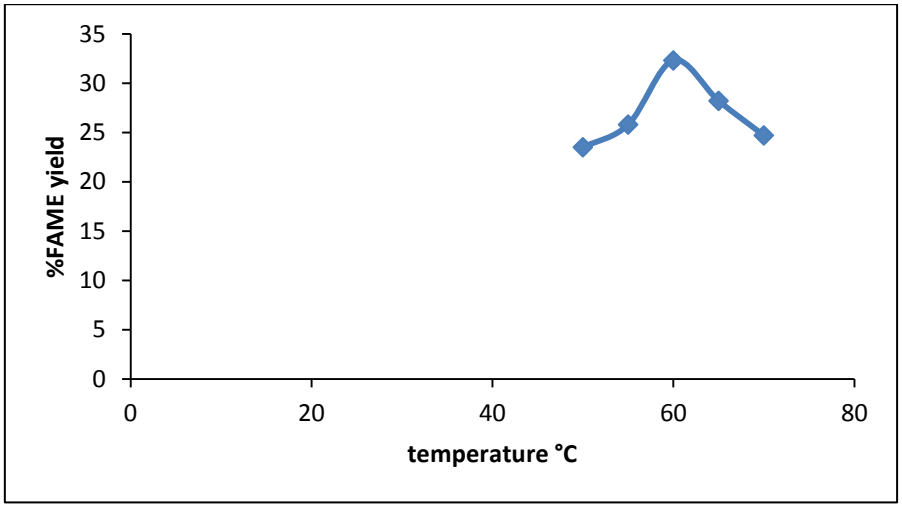


Figure 4.4: Effect of reaction temperature on conversion of lipid to biodiesel

4.2.3 Effect of Reaction Time

The effect of reaction time was varied from 1-4 hours, while maintaining methanol: lipid ratio 11:1, reaction temperature of 60 °C and fixed amount of catalyst (2.5 wt %). Figure 4.5 shows the interference of reaction time in conversion of lipid to biodiesel. The increase in fatty acid esters conversion was observed when there is an increase in reaction time. The

reaction is slow at the beginning due to dispersion of alcohol and lipid. After that the reaction proceeds very fast. The maximum biodiesel yield of 33.2% was achieved in 3 hours and thereafter the conversion decreased. It may possibly due to reverse reaction and formation of fatty acids ((Encinar et al., 2012; Eevera et al., 2009). However, by extending the reaction time to 4 hours and 5 hours, biodiesel yield probably starts decreasing to give a yield of 28.6 % and 22.5%, respectively. This may be due to the fact that reaction has reached equilibrium. Also longer reaction time may lead to reversible reaction of transesterification resulting in loss of esters as well as soap formation (Mathiyazhagan and Ganapathi, 2011; Jagadale and Jugulkar, 2012). Table 4.7 compares the percentage of biodiesel yield obtained from food waste with other raw materials

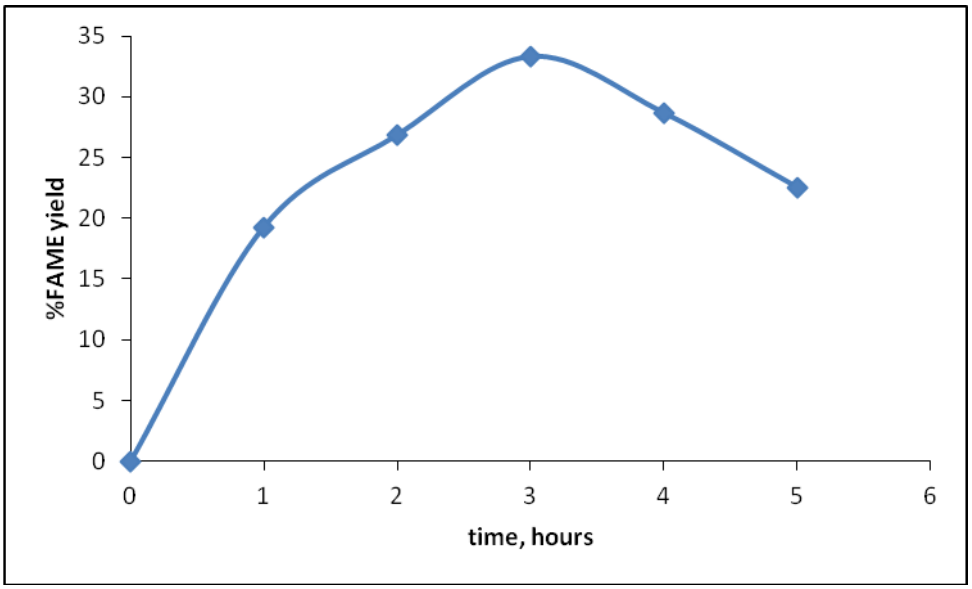


Figure 4.5: Effect of reaction time on conversion of lipid to biodiesel

Comparison of biodiesel yield obtained from different raw materials with present study has been discussed in table 4.5. It was observed that the raw materials having very high biodiesel yield is costlier because of raw material cost. Though food waste biodiesel yield is less, its raw material cost is zero. Biodiesel obtained from food waste can be blended with diesel to the give same efficiency as that of raw materials having higher percentage of yield.

Table 4.5: Comparison of biodiesel yield obtained from different raw materials with present study

S.N.	Researchers	Raw materials	Percentage of biodiesel yield (%)
1.	Rao et al., 2015	Pongamia pinnata oil	75
2.	Yahya et al., 2016	Waste cooking oil	65
3.	Raqeeb and bhargavi, 2015	Waste cooking oil	80
4.	Siddhique and Rohani, 2011	Waste water sludge	51.39
5.	Olkiewicz et al., 2016	Sewage sludge	65
6.	Nowicki et al., 2016	Rapeseed oil	54.2
7.	Torres et al., 2014	Soybean oil	98.3
8.	Veillette et al., 2017	Microalgae	84
9.	Rodrigues et al., 2016	Jatropha oil	80.5
10.	Present study	Food waste	32.3

4.3 FAME ANALYSIS

The biodiesel produced after transesterification was analyzed in GC-FID for fatty acid methyl ester (FAME) analysis.

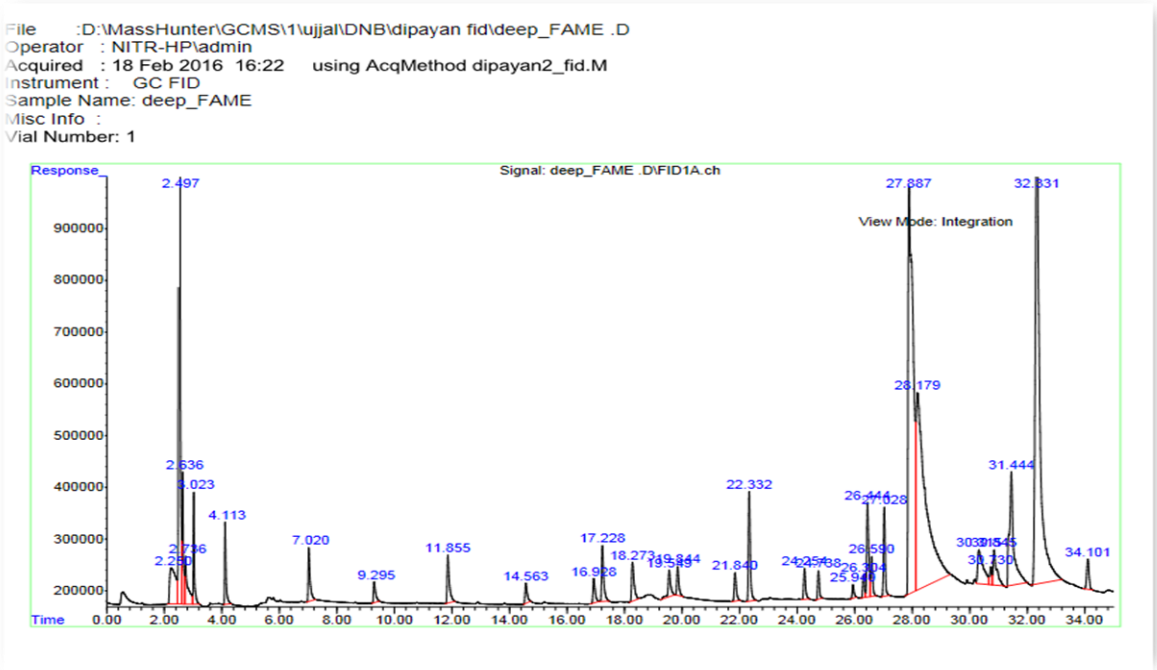


Figure 4.6: GC-FID analysis of FAME standard

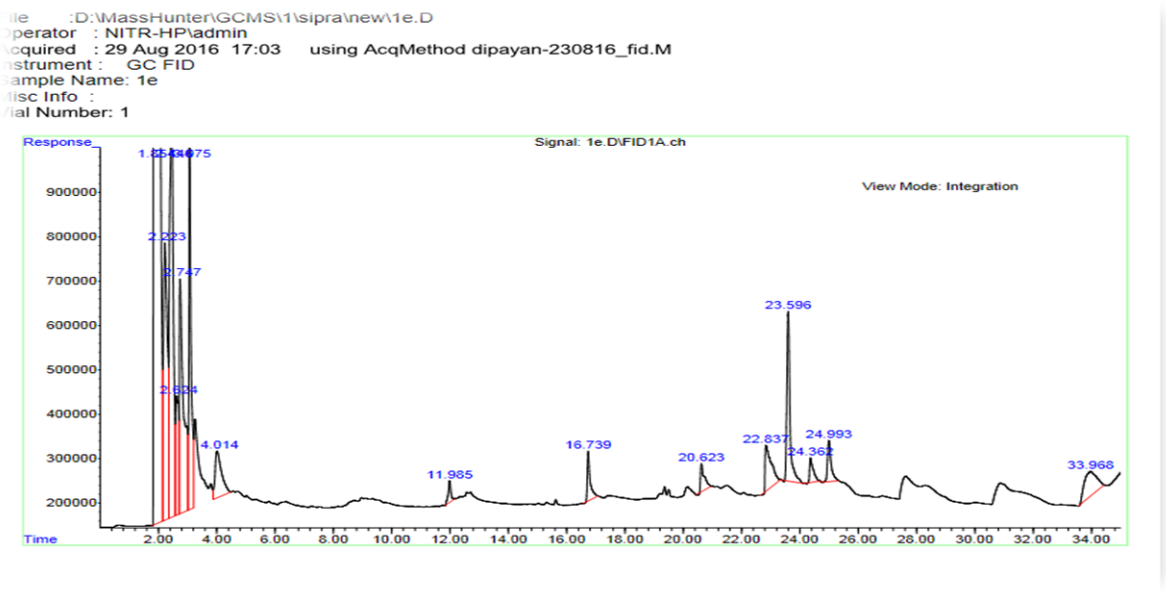


Figure 4.7: GC-FID FAME analysis of food waste

The compounds identified from the peaks of GC-FID by comparing with the standards are discussed in table 4.6. The identities of individual FAME were confirmed through the analysis of FAME standard. The fuel properties of fatty esters depend on chain length, number and position of double bond.

Table 4.6: Fatty acid profile of biodiesel from food waste

S.N.	**Retention time**	**FAME**	**wt%**
1.	4.014	Caproic acid methyl ester (C6:0)	17.64
2.	11.985	Lauric acid methyl ester (C12:0)	4.04
3.	16.739	Mystric acid methyl ester (C14:0)	9.23
4.	22.837	Palmitic acid methyl ester (C16:0)	21.23
5.	23.596	Palmitoleic acid methyl ester (C16:1)	11.06
6.	24.362	Stearic acid methyl ester (C18:0)	19.19
7.	24.993	Oleic acid methyl ester (C18:1)	15.67

Biodiesel components were confirmed using gas chromatography. Gas chromatography analysis of the fatty acid methyl ester (FAME) was performed in order to determine the fatty acid components of lipid extracted from food waste. The graph in figure 4.7 and table 4.5 shows each peak representing individual FAME components such as caproic acid (17.64%), lauric acid (4.04%), mystric acid (9.23%), palmitic acid (21.23%), palmitoleic acid (11.06%), stearic acid (19.19%), oleic acid (15.67%). The results proved that lipid extracted from food

waste proved to be suitable for biodiesel production; converting lipids to fatty acid methyl ester (FAME). Saturated components such as mystric acid (C14:0), palmitic acid (C16:0), stearic acid (C18:0) have higher cetane numbers and are less prone to oxidation than unsaturated compounds but they tend to crystallize at unacceptably high temperature. This could be solved by the presence of branched-chain and hydroxyl fatty acid methyl esters (Olkiewicz et al., 2014). As saturated fatty acids is more than unsaturated fatty acids cetane no. (CN) will be high for biodiesel. These reports were given by Harrington (1986); van Gerpen (1996); Knothe et al. (2003). Saturated fatty acids also have high melting point. As unsaturated fatty acids are less, biodiesel obtained from food waste will not undergo auto oxidation. The rate of oxidation depends on the number and location of allylic methylene positions (adjacent to the double bond) on the backbone of olefinic compounds. With respect to long chain fatty acid methyl ester (FAME), polyunsaturated esters are significantly more vulnerable to autoxidation than monounsaturated esters because they contain more allylic methylene positions. For high quality of biodiesel both saturated and unsaturated of FAME should have optimal balance.

It was found that saturated fatty acids dominate in FAME analysis of lipid. Due to saturated components, biodiesel produced from food waste will undergo cold flow problem. Several studies were conducted by various researchers to improve cold flow properties of biodiesel such as use of additives to reduce intermolecular bond, decrease the crystalline temperature and combining biodiesel with petro diesel, as well as the use of thermal cracking process, ozonation technique and winterization technique to reduce the concentration of saturated fatty acid esters. However, specific method or additive that can improve cold flow behavior of all types of biodiesel is not available. Cold flow enhancers are used to improve the cold flow properties of biodiesel (Monirul et al., 2015). According to Refaat (2009), length of fatty acids chains as well as its composition plays a substantial role in cold flow properties of biodiesel. National biodiesel board (2013) also stated that properties of biodiesel depends on the raw material (type of grease, fat or oil) from which they are produced and strong factor to determine the amount of saturated fat. Krishna and Butcher (2008) suggested to improve the cold flow properties, biodiesel should be blended with petro diesel. This will help to reduce pour point and saturated fatty acids in biodiesel. Dunn (2009) suggested that to improve cold flow problem, properties of biodiesel can be improved by modifying its fatty acid profile to remove high melting components. Kim et al. (2012) examined the cold performance of biodiesel blends in a car and a light duty truck at −16°C and −20°C, using jatropha, soybean, palm, rapeseed, cottonseed, and waste cooking oil derived biodiesels

mixed in different fractions (B5, B10 and B20). They observed that the presence of unsaturated structures and the hydrocarbon chain length has marked effect on the low-temperature properties of biodiesel as the start-ability and drivability of the car with all B5 blends were generally good at −20 °C. While the B10 and the B20 blend of palm biodiesel failed at −20°C and at −16 °C respectively in the start-ability test, the B10 and B20 blends of jatropha, soybean, rapeseed and waste cooking tended to be good at −20 °C in both tests. The addition of cold flow improvers coupled with blending, B1, B5, and B20 in most cases enhanced the cold flow properties of soybean and canola biodiesel (Conley, 2012). Jon Van Gerpen (2013) in another research also reported that blending improves cold flow properties of biodiesel, when he blended Tallow and soybean oil based biodiesel with diesel.

4.4 BIODIESEL PROPERTIES

The physico-chemical properties of biodiesel were performed after FAME analysis to check properties by comparing with ASTM, EN and Indian standard which is summarized in table 4.7.

Table 4.7: Comparison of biodiesel properties with ASTM, EN and IS standard

S.N.	Properties	ASTM D6751	EN14214	IS 15607 (2005)	Obtained value
1.	Density at 15°C (kg/m^3)	875-900	860-900	860-900	872
2.	Kinematic viscosity (mm^2/s)	1.9-6.0	3.5-5.0	2.5-6.0	2.2
3.	Acid value (mg of KOH/g), Max	0.5	0.5	0.5	0.63
4.	Pour point (ºC)	-3 to 12	-	-	7
5.	Calorific value (MJ/kg)	-	35	-	31.38
6.	Metals (Na+K) (mg/kg)	5	5	-	1.5
7.	Metals (Ca+Mg) (mg/kg)	5	5	-	1.25
8.	Ash content, percent by mass, Max	0.02	0.02	0.02	0.0072
9.	Cloud point (ºC)	-	-	-	12
10.	Flash point (ºC), Min	130	101	120	164

Density of biodiesel produced from food waste is found to be 872 kg/m^3 which is within the limit according to ASTM, EN and IS standard. The densities of biodiesel are generally higher than that of petro based diesel fuel. The values depend on their fatty acid composition as well as on their purity. Density increases with decrease in chain length of fatty acid and

increasing number of double bonds or can be decreased by the presence of low density contaminants such as methanol.

Viscosity is a measure of resistance of flow of a fluid. It is due internal friction of molecules in fluid. High viscosity means high resistance to flow while low viscosity means low resistance to flow. One of the most important properties of biodiesel is kinematic viscosity. Higher viscosity of biodiesel enhances fuel spray penetration and improves air fuel mixing as discussed by some researchers like Lin et al. (2009), Öner and Altun (2009); Monyem et al. (2001). However, few researchers like Aydin and Bayindir (2010); Utlu and Koc (2008) thought that higher viscosity results in power losses, because higher viscosity decreases combustion efficiency due to bad fuel injection automization. The kinematic viscosity of biodiesel is found to be 2.2 mm^2/s which in the range of 1.9-6.0 according to ASTM D 6751 according to test method D445. This implies that biodiesel enhanced fluidity of fuel for diesel engine and good spray pattern that would generate across the combustion chamber, allowing proper mixing with air (Gandure et al., 2013). Emil et al. (2009) has quoted that Viscosity increased with molecular weight but decreased with increasing unsaturated level and temperature. For direct application in engine biodiesel should have low viscosities. High viscosity of biodiesel fuel is not suitable for use directly on fuel engine because it may cause operational problem such as carbon deposits, oil ring sticking, thickening and gelling of lubricant oil.

Acid value is the measure of amount of carboxylic acid groups in fatty acids. It is used to quantify the amount of acid present in biodiesel. The acid value for biodiesel is 0.63mg KOH/g and it exceeds the EN 14214 and ASTM D6751 standard according to which acid value should not exceed the value 0.5mg KOH/g. as triglycerides present in oil and fats converted to fatty acids causing an increase in acid number. These fatty acids produced may corrode automobile parts, vehicle engines and fuel tanks.

Pour point is the lowest temperature at which oil starts to melt or pour. It is the measure of ability of a fuel to operate under cold weather conditions. The pour point of biodiesel is found to be 7°C which is within the range of ASTM D6751 (-3 to 12 °C). During cold weather biodiesel causes fuel starvation and operational problem which cease the fuel flow in engine. It also causes pumping problem in engine in cold climate (Dwivedi and Sharma, 2014).

Calorific value is the measurement of heat or energy produced and is measured either as gross calorific value or net calorific value. The gross calorific value of biodiesel measured by bomb calorimeter is found to be 31.38MJ/kg which is less than the standard i.e., 35MJ/kg

according to EN 14214 standard. Calorific value of diesel fuel is 45.5MJ/kg. Less calorific value means more fuel consumption. To solve this problem biodiesel can be blended with diesel fuel to improve its calorific value.

Metal ions are introduced into the biodiesel fuel during production process. Sodium and potassium are associated with the formation of ash within the engine. Calcium soaps are responsible for injection pump sticking (Mittelbach, 2000). Alkali metals (Na, K, Ca and Mg) can form sediments and cause injector failures. If metal contamination can occur, metal deactivators can be used to chelate transition and inhibit catalytic oxidation and polymerization effect. Alkali metals could also form soap and cause insoluble of diesel blends. Alkali metals of (Na+K) were found to 1.5 mg/kg and (Ca+Mg) were found to be 1.25mg/kg which is within the range of 5mg/kg according to standard EN 14538.

Flash point is the minimum temperature at which vapor given off by a fuel when heated will flash with a test flame held above the surface without catching fire and plays a vital role in fire hazard of fuel. It gives the idea about the boiling point of liquid fuel, volatility and explosive hazards. Flash point (164 °C) of biodiesel obtained from food waste is within the limit according to ASTM, EN and IS standard.

Cloud point of any fuel is defined as the temperature at which crystals form in oil which it is cooled at specific temperature. Using fuel at low temperature causes fuel filter clogging because of crystal formation. As the biodiesel starts to freeze, it will form crystals that start clumping together. They will become visible as cloudy biodiesel when they start to grow larger. Cloudy biodiesel can easily pass through filters and can be pumped with no problem. When the biodiesel starts to solidify with temperature and form crystals it will create problem in plug filters. Cloud point of obtained biodiesel is 12 °C i.e. below this temperature biodiesel will form a cloudy appearance.

Ash point describes the amount of inorganic contaminants such as abrasive solids, catalyst residue, soluble metals contained in fuel. These compounds are oxidized during combustion process to form ash which is connected with engine deposits and filter plugging (Mittelbach, 1996). Ash content of biodiesel is found to be 0.0072 which is within the standard value (0.02).

4.5 CHEMICAL CHARACTERIZATION OF RESIDUE LEFT AFTER LIPID EXTRACTION

Characterization of residue left after lipid extraction is done to identify the chemical species and its further reuse technique. The characterization of residue helps to ensure that they meet

the specified limit mentioned in standard prior to land disposal or its further reuse which are discussed in table 4.8. Total hardness is caused due to calcium and magnesium. As total hardness is very high indicating high concentration of calcium and magnesium. Calcium is considered as secondary nutrient for plant growth. Calcium uptake by plant is passive and does not require energy.

Adsorbed sodium might cause the soil to crack when dry and swell up when wet. Calcium replaces the adsorbed sodium and prevents damages to soil structure. Magnesium is also an important plant nutrient. One of the important functions of magnesium is that it is a building block of chlorophyll which makes leave appear green. Deficiency of magnesium result in chlorosis of plant leaves as it moves readily from lower to upper parts of plant, the deficiency first appear on lower leaves. Calcium and magnesium present in food waste can be used for plant nutrient when disposed on land.

The ideal soil pH is close to neutral and is considered to fall within the range from slightly acidic pH 6.5 to slightly alkaline pH of 7.5. It is found that most plant nutrients are available to plants within the pH range of 6.5 to 8.0 and this range of pH is very useful for plant root growth. A pH range of approximately 6 to 7 promotes the most ready availability of plant nutrients. The above parameters discussed can be helpful to minimize handling and disposal problem for food waste. Some parameters like pH, total hardness (Calcium and Magnesium) can be used as nutrients for plant growth.

Table 4.8: Concentration value of different chemical parameters

S.N.	**Parameter**	**Unit**	**Concentration**	**Permissible limit for plant nutrients according to WHO**
1.	Total hardness	mg/kg	830.0	-
2.	pH	-	6.72	6.5-8.0
3.	Calcium	mg/kg	22.71	-
4.	Magnesium	mg/kg	2.62	-
5.	Copper	mg/kg	0.65	10
6.	Zinc	mg/kg	2.35	50
7.	Chromium	mg/kg	0.98	1.30
8.	Iron	mg/kg	30.47	20
9.	Nickel	mg/kg	0.15	10

The term heavy metal refers to any metallic element that has relatively higher specific gravity (typically five times heavier than water) and is often toxic or poisionous even at low concentrations. This group of heavy metals includes lead (Pb), cadmium (Cd), nickel (Ni), cobalt (Co), iron (Fe), zinc (Zn), chromium (Cr), arsenic (As), silver (Ag) and the platinum

(Pt) group of elements (Farlex, 2005). Some heavy metals have bio-importance but bio-toxic effect on human health is of great concern (Mahurpawar, 2015). In the present study, the microwave digested samples are utilized for determination of metallic species by using atomic absorption spectrophotometer (AA200, Perkin Elmer). The elemental concentrations are summarized in table 4.10. Calcium (22.71 mg/kg) and zinc (2.35 mg/kg) dominate the metallic species.

Calcium is very essential in muscle contraction, building strong bones and teeth, blood clotting, nerve impulse, transmission, regulating heart beat and fluid balance within cells. Long term calcium deficiency can lead to oestoporosis in which the bone deteriorates and there is an increased risk of fractures. It is very essential for maintaining the tone and contractility of heart (Faryadi, 2012). Calcium is also helpful for plant growth as it act nutrients for plant. As calcium concentration is 22.71 mg/kg, pharmaceutical industry can extract calcium from food waste and reuse for medicinal purpose.

Magnesium is responsible for the activation of more than 300 enzymes in the body. People with magnesium deficiency are always tired, irritable, nervous, stiffness in the muscles and difficult to concentrate. Magnesium deficiency is actually linked to various pathological conditions. Magnesium is one of the most essential mineral in human body. Study indicates that there is a linkage between magnesium deficiency and chronic joint pains as magnesium deficiency causes muscle tension and spasm (Freeman, 1999). Although magnesium concentration (2.62 mg/kg) is less as compared to calcium (22.71 mg/kg) it can be used with calcium as a supplement.

Copper is a naturally-occurring metallic element and it occurs in soil at an average concentration of about 50 parts per million. Copper is found in all animals and plants and an essential nutrient for humans and animals even in trace quantity. The recommended daily allowance of copper for adults is 0.9 milligrams (mg). Permissible limit of copper for plant nutrient is 10 mg/kg and it is within the limit. Copper (0.65 mg/kg) present in food waste can be used for pharmaceutical as well as plant nutrients.

Zinc plays a vital role in the maintenance of immune functions and its deficiency affects multiple aspects of innate and adaptive immunity. The function of zinc is to help a plant to produce chlorophyll. Leaves get discolor when soil is deficient in zinc and plant growth is stopped. Zinc is an essential micronutrient that affects several metabolic processes of plants and has a long biological half-life (Asati et al., 2016). Zinc (2.35 mg/kg) is within permissible limit (50 mg/kg) which can be used as nutrients for plants and medicine for human purpose.

Chromium (Cr) compounds are highly toxic to plants and affects plant growth and

development (Davies et al., 2002). As chromium (0.98 mg/kg) concentration in the present study is less it will not affect plant and human health directly.

Iron is an essential micronutrient for all living organisms as it plays a vital role in metabolic. It plays a significant role for plants in basic biological processes such as photosynthesis, chlorophyll synthesis, respiration, nitrogen fixation, uptake mechanisms (Kim and Rees, 1992). Average iron concentration obtained in the food waste samples is 30.47 mg/kg exceeding the permissible limit (20 mg/kg) for plant nutrients. Also it can be used for medicinal purpose by drug industry for preparing iron tablets.

Nickel compound occur in environment at very low levels. Foodstuffs usually contain small amounts of nickel. Consumption of large amount of nickel directly or indirectly results in lung cancer, nose cancer and prostate cancer. Sensitive individuals may develop a skin disease known as nickel itch when exposed to nickel and its compound. Nickel (0.15 mg/kg) concentration is very less indicating its negligible effect on human and plants. Also it can be use as plant nutrients.

The extracted waste after lipid extraction can be used by drug industry for medicinal purpose and also by fertilizer industry for plant nutrients. This will help to reduce waste disposal and handling problem after biodiesel production. This innovative approach of utilizing extracted waste further for human purpose will be helpful for sustainable development and reduce environmental pollution.

CHAPTER 5

CONCLUSION

5. CONCLUSION

To overcome the fossil fuel crisis and its environmental pollution, renewable resources are preferred to replace nonrenewable resources. An alternative way to meet fuel demand is to convert renewable resources with zero value raw material cost to highly value added product i.e., biodiesel. In this research work an initiative has been taken to reuse kitchen food waste for the production of biodiesel. Food waste samples collected from CVR hostel of National Institute of Technology, Rourkela has been collected and dried by various drying methods i.e., oven drying (55 ^{o}C, 70 ^{o}C and 105 ^{o}C), freeze drying (-4 ^{o}C) and sun drying. Dewatering of sample is a vital step because water present in the sample may surround the particle and prevent penetration of solvent into the organic compounds. Oven drying method at 105 °C is selected as the optimum temperature for drying method. Lipid has been extracted from food waste using soxhlet apparatus and magnetic stirrer by using methanol:chloroform (2:1) as solvent according to Bligh dyer's method. For optimized lipid yield, sequential extraction was carried out at different ratios (1:1, 1:2, 1:4, 3:1 and 4:1) during different extraction time (20, 40, 60, 80, 100 min) for 10 consecutive extraction stages. For optimized production with minimum solvent consumption of lipid yield, 2:1 ratio gave best yield of 37.3% in 60 minute at eighth stage. Also, ratio 2:1 give almost same yield with less solvent consumption as compared to ratios 3:1 and 4:1. Hence, ratio 2:1 is considered economical giving maximum lipid yield of 37.3% in 60 minute at eighth stage. The extracted lipids were further been analyzed in GC-MS to identify the presence of organic acids. Saturated fatty acids identified in lipid include caproic acid (6:0), lauric acid (12:0), mystric acid (14:0), palmitic acid (16:0) and stearic acid (17:0). Unsaturated fatty acid identified in lipid is oleic acid (18:0). Acid and base transesterification is adopted for conversion of lipid extracted from food waste to biodiesel. The produced biodiesel has been further characterized in GC-FID to identify the concentration of fatty acid methyl ester (FAME). The produced biodiesel is characterized and compared with national (IS 15607) and international standards (EN 14214 and ASTM D6751). Density (872 kg/m^3), kinematic viscosity (2.2 mm^2/s), pour point (7°C), ash content (0.0072) alkali metals (Na+K) (1.5 mg/kg) and (Ca+Mg) (1.25mg/kg) of biodiesel produced from food waste is found to be within the limit according to ASTM, EN and IS standards. The calorific value of biodiesel is found to be 31.38MJ/kg which is less than the standard (35MJ/kg) according to EN 14214 standard. Cloud point of obtained biodiesel is 12 °C i.e., below this temperature biodiesel will form a cloudy appearance. Thus, it is found that food waste can be a better alternative source for biodiesel production. The produced biodiesel is

cost effective than conventional diesel fuel because of zero cost raw material. Metallic characterization (Ca, Mg, Cu, Zn, Cr, Fe, Ni) have also been performed of residue left after lipid extracted from food waste to identify its other possible reuse techniques. Calcium (20.36 mg/kg), iron (30.84 mg/kg), magnesium (3.00 mg/kg) and chromium (1.28 mg/kg) dominate the metallic species. The residue thus can be further reused by pharmaceutical industries after extracting these metals and also as soil and plant nutrients. This reuse technique is an innovative sustainable approach to minimize fuel crisis, green house emission and waste disposal problems.

5.1 SCOPE FOR FURTHER RESEARCH

- More varieties of wastes to be included and then compare the result for optimized yield of biodiesel.
- Detailed study on construction cost and other cost to be incorporated to find the total cost of the produced biodiesel.
- Study on cost-benefit analysis.

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